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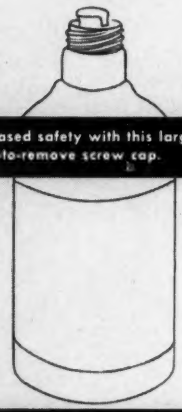

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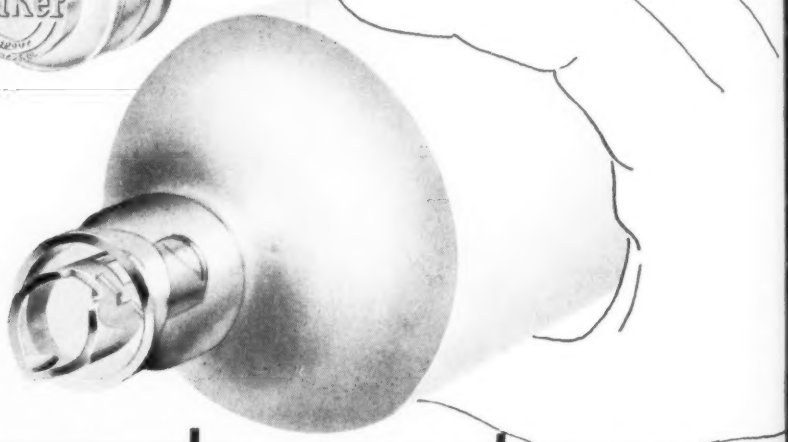

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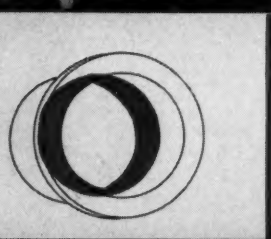

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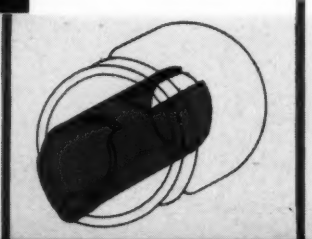

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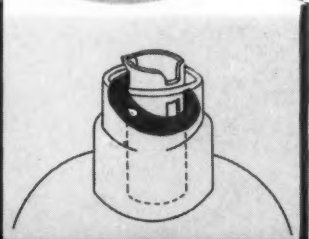

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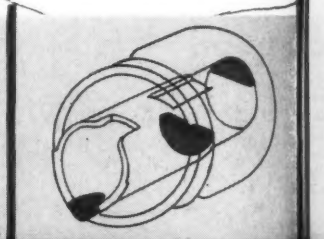

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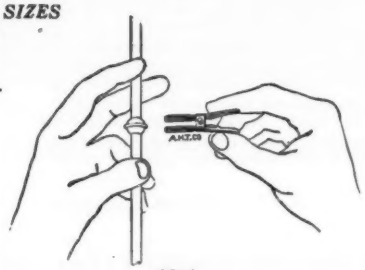

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Fig. 2, showing method of attaching to joint. Parts held securely by spring upon release of finger pressure.

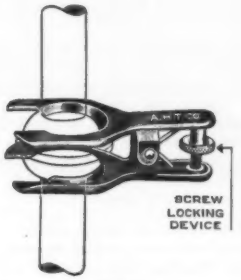

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Fig. 3, showing appearance of Sizes 28 to 75, incl., with locking device screwed into position.

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Fig. 4, showing appearance of Size 102 and method of fastening in position.

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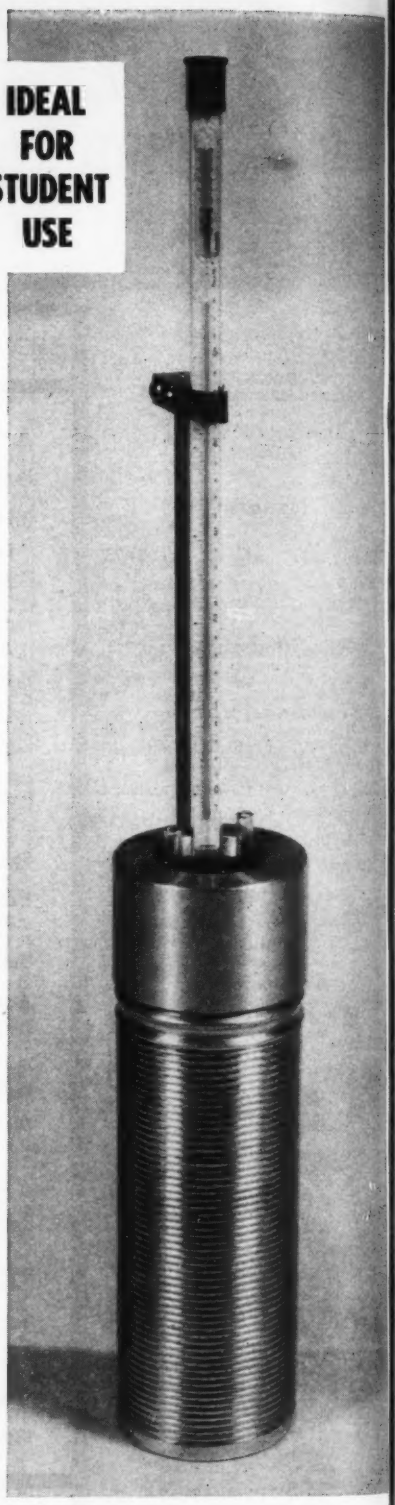

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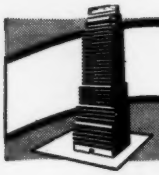

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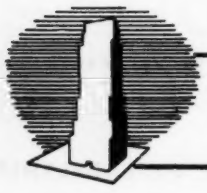

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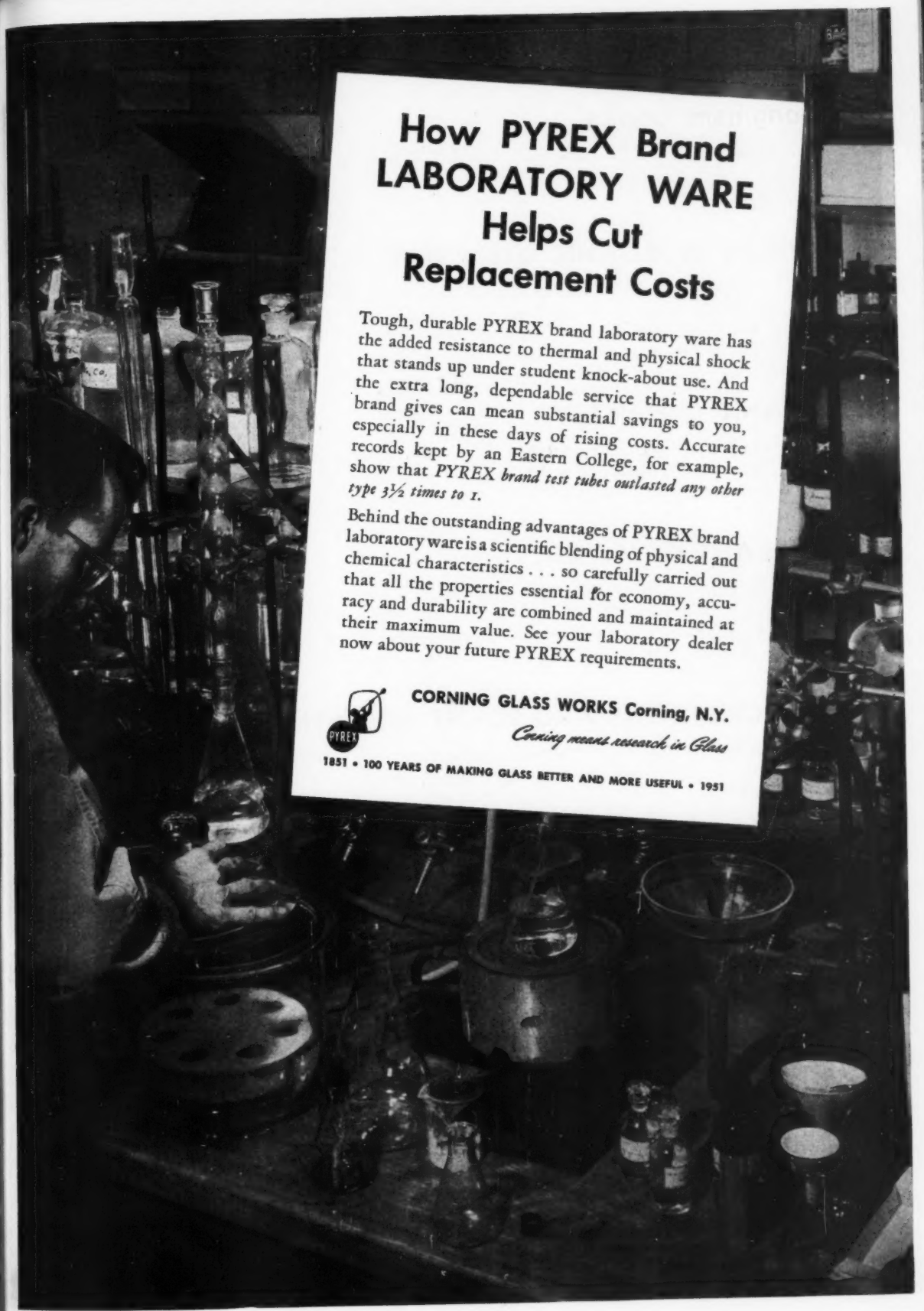
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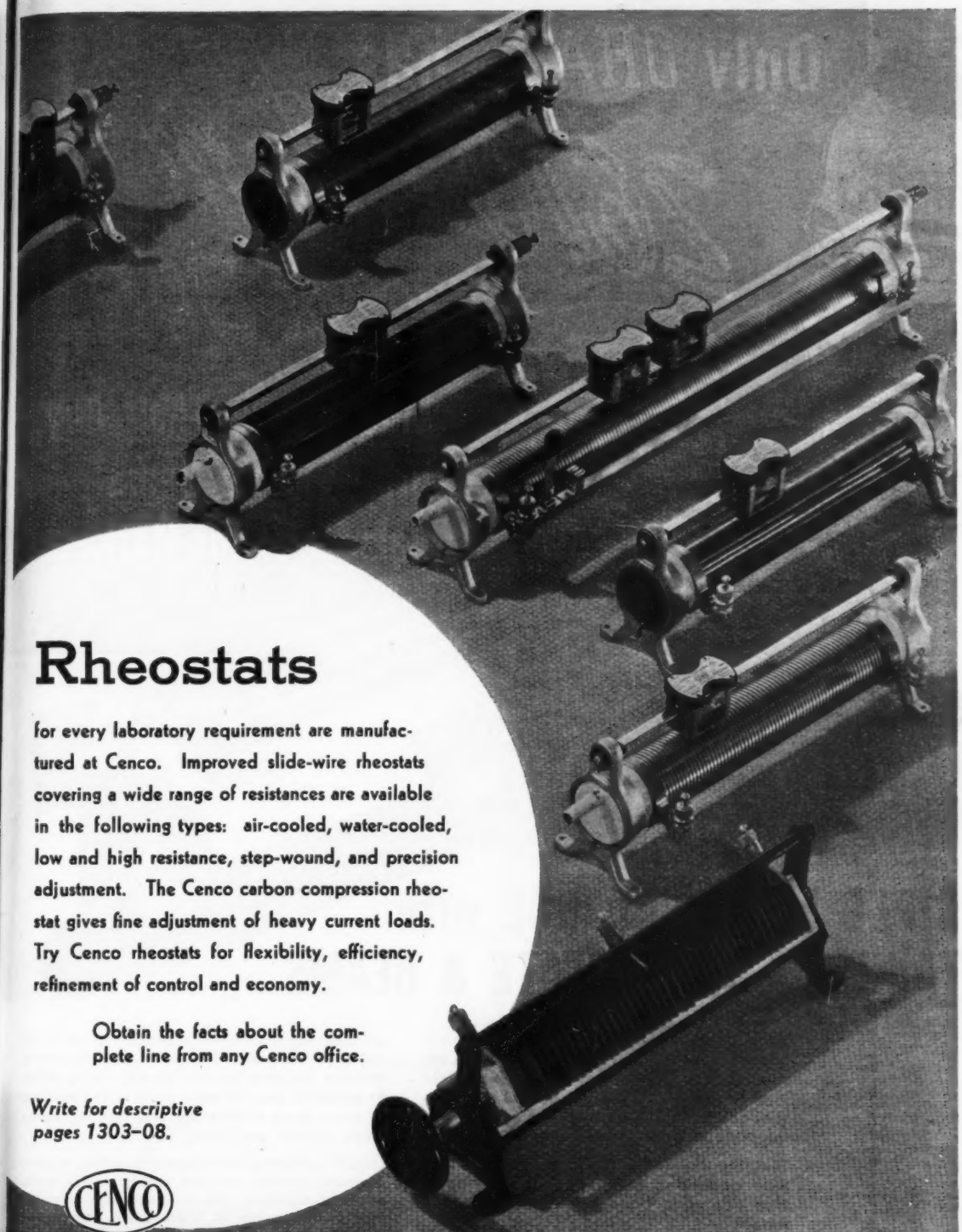

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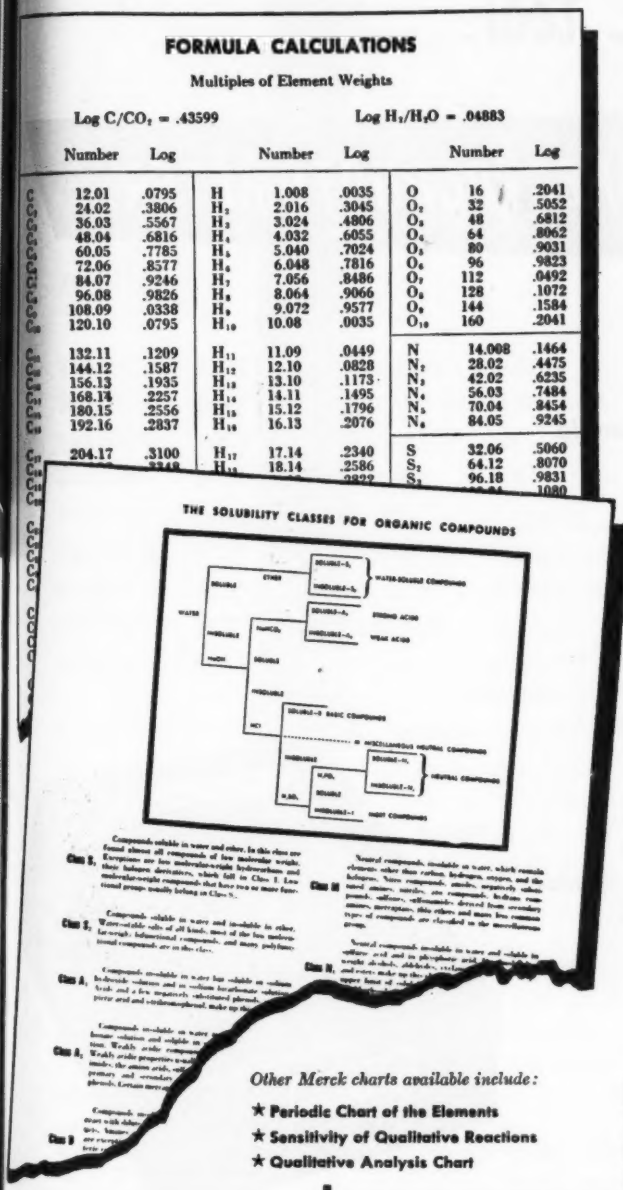

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24.02	.3806	H_2	2.016	.3045	O_2	32	.5052
36.03	.5567	H_3	3.024	.4806	O_3	48	.6812
48.04	.6816	H_4	4.032	.6055	O_4	64	.8062
60.05	.7785	H_5	5.040	.7024	O_5	80	.9031
72.06	.8577	H_6	6.048	.7816	O_6	96	.9823
84.07	.9246	H_7	7.056	.8486	O_7	112	.0492
96.08	.9826	H_8	8.064	.9066	O_8	128	.1072
108.09	.0338	H_9	9.072	.9577	O_9	144	.1584
120.10	.0795	H_{10}	10.08	.0035	O_{10}	160	.2041
132.11	.1209	H_{11}	11.09	.0449	N	14.008	.1464
144.12	.1587	H_{12}	12.10	.0828	N_2	28.02	.4475
156.13	.1935	H_{13}	13.10	.1173	N_3	42.02	.6235
168.14	.2257	H_{14}	14.11	.1495	N_4	56.03	.7484
180.15	.2556	H_{15}	15.12	.1796	N_5	70.04	.8454
192.16	.2837	H_{16}	16.13	.2076	N_6	84.05	.9245
204.17	.3100	H_{17}	17.14	.2340	S	32.06	.5060
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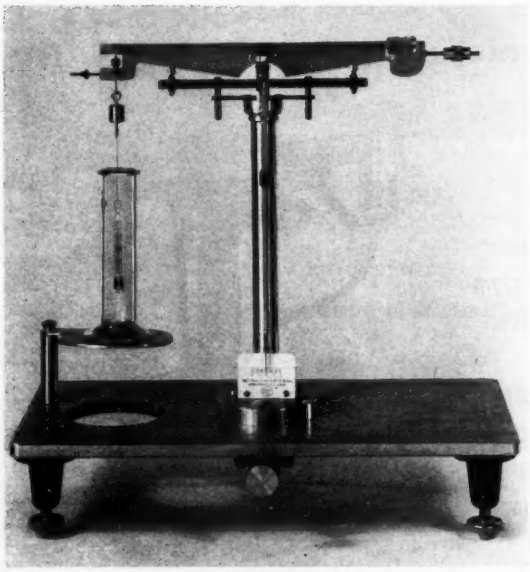

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(See page 62)

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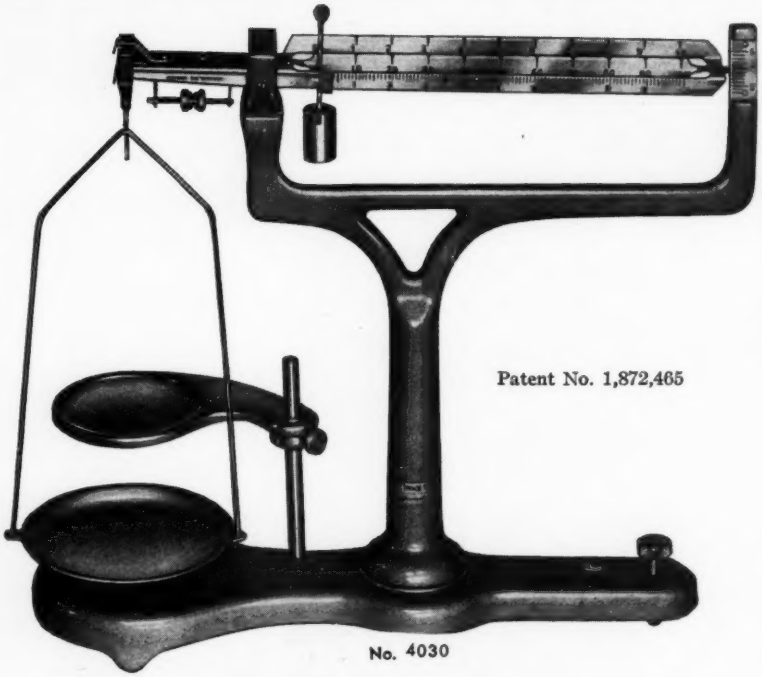

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Editor's Outlook

One frequently meets the opinion that the standard of teaching in the secondary schools is very much superior to that in the colleges; I have even heard it suggested that teaching is best of all in the elementary school. Of course, all this assumes adequate sampling and perhaps disregard of the "exceptions."

This is not to be wondered at, perchance. The high-school teacher has practically nothing to do *but* teach (in terms of quantity, unfortunately, plenty!) while the college teacher has—for better or worse—other things on his mind. Not the least of these is often the commendable yearning desire to contribute to the intellectual stockpile of his chosen field of knowledge, which makes him jealous of the time spent in pounding bright spikes of wisdom into the resistant oak of calow minds. Indeed, as is well known, there is little incentive to good teaching in our large universities, first, because of the crushing effect of the waves upon waves of students, and second, because of the more tangible rewards and flattering consequences of "creative scholarship" or "research." Very likely there will be those—particularly from smaller colleges—who will jump to the defense of college teaching. Indeed, I hope they will; college teaching needs defenders—I've long been one myself.

As one goes up the educational ladder, teaching rather seems to become worse, until one reaches the top, where, in the graduate school, "teaching" virtually ceases to exist. Still, this seems to be unimportant, for here *learning* is at its best. The graduate student doesn't—and shouldn't—rely upon teaching. If he is successful he is self-taught, independent. Without doubt, this is the ultimate goal of all teaching; at this stage teaching methods and all the pedagogical paraphernalia are of no consequence. Formal instruction still exists, to be sure, and usually proceeds by the much-bewailed "lecture" formula, seldom enlivened by demonstrations or audio-visual aids. Still it is reasonably effective, perhaps in spite of, rather than because of, the method. However, the real learning here comes from quite different sources: reading, long literature searches, attendance at and active participation in seminars and colloquia, discussion and arguments with fellow students and professors, laboratory "bull-sessions" far into the night. And lastly, the acme of all—learning by doing. Here research is absorbed *into* teaching, and when the student is initiated into this mystery he really belongs to the cult.

It is not all plane sailing; students can fail here, even more disastrously than at lower levels. But if so, they cannot blame it upon poor teaching; they alone are responsible. On the other hand, this state of affairs cannot come into being of its own accord. One or two graduate students working in an isolated laboratory do not make a graduate institution; an *esprit de corps* is the most important requirement, and this has to be built up around a suitable nucleus. Anyone who has lived in such an environment knows that it is not an academic ivory tower.

The answer to the undergraduate problem—if it really is a problem—is to bring up into this level some of the enlightened teaching techniques of the secondary school, and to bring down from the graduate school some of the incentive to self-teaching and the independence from intellectual coercion. Perhaps such a program will bog down under the handicaps placed upon both students and teachers by their preoccupation, distraction, or mental inertia, but it is worth a try.

A METHOD OF ESTIMATING THE BOILING POINTS OF ORGANIC LIQUIDS

D. E. PEARSON
Vanderbilt University, Nashville, Tennessee

THE relationship of molecular structure to the boiling point of an organic liquid is discussed in this paper with the view of extending the enlightening contributions of McElvain (*1*) and Shriner and Fuson (*2*). The approach is simple enough and seemingly comprehensive enough to enable the interested reader to predict the boiling point of most classes of organic liquids with an accuracy of ±5°. If such is the case, the method must necessarily be consistent and cognizant of most of the important factors of molecular structure which influence boiling points.

The method remotely resembles that of Kinney (*3*) to the extent that a boiling point estimation of an organic liquid is based on the boiling point of the parent hydrocarbon to which an increment factor is added for the particular functional group.

A. *The Parent Straight-Chain Hydrocarbons*

Table 1 shows that the increment gradually decreases with increasing molecular weight, as to be expected for a proportional increment. Though it is not practical to continue beyond C_{13} or below C_4, an estimation can be made if desired. For example, the boiling point of propane must differ from butane by at least an increment of −35° and possibly another estimated −5°. The calculated boiling point of propane, therefore, is −40°, and it is actually found to be −42°. The C_{14} hydrocarbon must differ from the C_{13} hydrocarbon by an increment of 20° or less. The calculated boiling point of C_{14} is 255° and is actually 250°. At this point, an increment of 15° should be used and appears to be the limiting increment in further extensions. Obviously, considering both experimental determinations and the rough calculations, good correlation is not to be expected in or beyond this region of high molecular weight.

TABLE 1
Boiling Points of Paraffin Hydrocarbons (Straight Chain)

Hydrocarbon	*Rounded Figure*[a]	*Found*	*Increment*
C_4	0	−0.5	
			35
C_5	35	36	
			35
C_6	70	69	
			30
C_7	100	98.4	
			25
C_8	125	126	
			25
C_9	150	151	
			25
C_{10}	175	174	
			20
C_{11}	195	196	
			20
C_{12}	215	215	
			20
C_{13}	235	235	

[a] Memory is aided by noting that C_4, C_7, and C_{11} are 0°, 100°, and 200° (nearly).

The difference in empirical formula between the paraffin and unsaturated hydrocarbon molecule is two hydrogen atoms (or a multiple thereof). Since the small hydrogen atom has little effect on the volume and the density of the individual molecule (two factors which affect the boiling point), the boiling points of straight-chain olefins and acetylenes are found to be the same or only slightly less than those of the corresponding paraffin hydrocarbons.[1]

B. *The Effect of Branching and Ring Formation*

1. *For a single or double branching, subtract 10° from the boiling point of the parent hydrocarbon.* This increment is smaller, the closer to the center of the chain the branched group is attached.

2. *Two branches on a single carbon atom have a double effect—hence subtract 20°.* This increment also becomes less as the double branch approaches the middle of the chain.

3. *For a six-membered ring, whether aromatic or alicyclic, add 10° to the boiling point of the parent hydrocarbon.*

The above corrections are consistent with the conclusions that branching the chain decreases the *effective* volume of the individual molecule and that ring formation increases the *effective* volume. For example, pentane must have an elongated, "zig-zag" shape which together with its motion affords many more collision opportunities than the ball-shaped neopentane molecule, and, as a consequence, pentane (b. p. 35°) finds it more difficult to escape from the liquid than does neopentane (b. p. 10°). As another example, cyclohexane, like the doughnut, encloses space and again offers more collision opportunities than hexane. Thus, cyclohexane (b. p. 81°) does not as readily escape from the liquid as does hexane (b. p. 70°). The explanation for the effect of cyclization is further strengthened by observation that the increment, +10° for six-membered rings, increases with enlargement of the ring system. This is

[1] Space does not permit the inclusion of tables of illustration. It is left to the reader to check the general rules given.

to be expected if more space is enclosed within the ring.[2] It will be noted in checking the above system of predicting boiling points that a branched chain connected directly to a double-bond carbon does not lower the boiling point.

C. *Functional Group Equivalents (Non-associated Liquids)*

It is implied in Table 2 that the substitution of another atom for a carbon atom in the organic molecule affects the boiling point to the extent that the atomic "density" and volume of that atom is related to the carbon atom, or, more properly, the methylene group. The above is true provided the bond angle attachment of the new group is approximately the same as the comparative carbon attachment.

TABLE 2

Class	*Group*	*Methylene Equivalent*
Chloride	Cl	2+
Bromide	Br	3
Iodide	I	4
Ether	O	1−
Ester, anhydride, or acid chloride	O	1 (for each oxygen)
Sulfide	S	3
Mercaptan	SH	3

The table is readily applied in one's mind as follows: butyl bromide has four carbons plus one bromine atom, or the equivalent of three "methylenes"; four plus three methylenes is equivalent to the boiling point of heptane (100°). It is observed that the predicted boiling points of halogen-containing liquids check the experimental values closely with the exception of *geminal* halogen compounds. The striking deviations of this group parallel other unusual chemical and physical properties.

Oxygen in an organic molecule is quite similar in density and volume to a methylene group. Therefore, provided no association takes place, an oxygen atom is merely replaced by a methylene group in predicting boiling points, *i. e.*, diethyl ether should boil at the same temperature as pentane, and butyl acetate at the same temperature as octane. It will be noted that, following this procedure, the ethers boil 5 to 10° lower than the predicted values. This is attributed to the fact that the ether molecule is slightly folded at the points of attachment to the oxygen atom, thus reducing the *effective* volume or collision possibilities.

It is rather surprising that mercaptans fall into the non-associated group. And, it should also be noted that the very low molecular weight esters show some association.

D. *Functional Group Equivalents (Associated Liquids)*

The methylene equivalents in this group include an association factor. The association factor, furthermore, is a minimum value, *i. e.*, the lower molecular weight alcohols, ketones, aldehydes, and amines will boil somewhat higher than predicted. However, as the molecular weight increases, the association factor levels off to the value given.

TABLE 3

Class	*Group*	*Methylene Equivalent*
Aldehyde or ketone	O	2
Alcohol	OH	3
Amine	NH_2, NH	2
Nitrile	N	3

Organic acids and amides also fall in this group. However, it is much easier to recall that formic acid boils at 100° and the addition of one methylene group increases the boiling point by 20°. In terms of the explanations previously used, this increment assumes that association is considerable with the low molecular weight acid (exactly a dimer for acetic acid) and diminishes with each additional methylene group at such a rate that the increment remains constant. Amides, on the other hand, are usually solids and are not considered here, though they are very appreciably associated. It is also questionable whether nitriles are classified correctly. They would be expected to associate through their active methylene groups, and, in fact, the predictions are quite good assigning a methylene equivalent of 3 to the nitrogen of the nitrile group. However, pivalonitrile (b. p. 105°) has no active hydrogen and still boils at the predicted temperature.

E. *The Effect of Resonance*

If an olefin, ester, ether, phenol, or carbonyl group is conjugated with an aromatic ring (or attached to a benzyl group) add +10° to the boiling point. This correction factor implies that the bond (or bonds) of the group are straightened and strengthened by the increased participation in resonance with the benzene ring. The straightening produces more collision possibilities. It is undoubtedly too simple an explanation to attribute all factors involved in the +10° correction to resonance alone, when the correction is applied to so many varied structures. Certainly, there are varying degrees of resonance within these structures which should require a varying increment. However, the fact remains that the correction factor works and that it applies only to those compounds with the properly conjugated systems. As an example, phenetole (b. p. 172°) is predicted to boil at 170°; calculation: $C_8 + O_1 \cong C_9 \cong 150° +$ 10° (ring structure) + 10° (resonance correction) = 170°. Other examples give better or almost as good agreement with the exception of aromatic aldehydes. Their higher-than-predicted boiling points imply greater resonance (or perhaps association).

The considerable deviation of aromatic amines from the predicted values can now be appreciated. Aniline (b. p. 183°) is predicted to boil at 145°. The resonance must not only strengthen and straighten the bond between the aromatic ring and nitrogen but must also activate the hydrogens attached to nitrogen so that

[2] In this connection, the reported boiling point (*4*) of cycloundecane (b. p., 184°C.) is undoubtedly much lower than its actual boiling point.

association is appreciably increased over that of ordinary aliphatic amines. Surprisingly enough, tertiary aromatic amines boil abnormally high also, which may indicate association through the *para* hydrogen atoms.

In conclusion, it is interesting to speculate on the state of water at its boiling point in accordance with the principles of this paper. The monomer should be equivalent to one, or slightly more than one, methylene group. Since water boils at 100°, which is equivalent to seven methylene groups, it is possibly an aggregate of six to seven molecules at 100° in the liquid state. Since water exists as a monomer in the gaseous state (*5*), a possible explanation for the large heat of vaporization is that considerable energy is utilized to accomplish the dissociation of this large aggregate simultaneously with its vaporization.

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PAUL PFEIFFER

RALPH E. OESPER
University of Cincinnati, Cincinnati, Ohio

Paul Pfeiffer, director emeritus of the Chemisches Institut at Bonn, creditably continued the Kekulé-Anschütz line, which made this university one of the eminent centers of chemical instruction and research. He was born on April 21, 1875, at Elberfeld. After a year under Kekulé, Pfeiffer transferred (1894) to Zurich, where he took his doctorate in 1897 under Alfred Werner.[1] Short periods were spent in Wilhelm Ostwald's laboratory in Leipzig and with Arthur Hantzsch at Würzburg. Dr. Pfeiffer habilitated at Zurich in 1901, and in 1908 was promoted to an associate professorship in theoretical chemistry. He was called to head the department at Rostock in 1916, and in 1919 exchanged this post for a similar one at Karlsruhe. He succeeded Richard Anschütz at Bonn in 1922. His retirement in 1947 did not close his active career; he still delivers lectures and conducts laboratory researches.

Professor Pfeiffer's scientific activities have been concerned especially with problems of complex chemistry. He discovered the isomerism phenomena in the complex compounds of chromium and developed the theory of additive salt formation. He made extensive advances toward clarifying the constitution of halochromic compounds, and proved the existence of isomeric salts (colorless and colored) in the case of aminoketones. He also studied the preparation and constitution of the neutral salt compounds of amino acids and polypeptides, quinhydrones and related compounds, and in addition elucidated a series of combinations of medicinals. He explained the constitution of nickel dimethylglyoxime, the copper complex in Fehling's solution, and tartar emetic. He prepared the inner complex calcium and magnesium salts of special amino acids (Trilon A and B), which are important with respect to water hardening. In 1915 he introduced the coordination concept into the theory of crystal structure.

Among his purely organic fields of study, special note should be made of the experiments aimed at clearing up the constitution of brasilin and hematoxylin; his contributions to the theory of the betaines; his investigations of the autoxidation phenomena among the anils of indandione; and the synthesis of the hitherto unknown β-chromanone. On the basis of complex chemistry, he developed a very simple procedure for the determination of the relative configuration of the natural α-amino acids, and it turned out that, with respect to its configuration, glucosamine is not related to the natural α-amino acids.

Professor Pfeiffer is the author of two widely known books: "Organische Molekülverbindungen" (1922, second edition 1927); and revision of A. Werner's "Neuere Anschauungen auf dem Gebiet der anorganischen Chemie" (1923).

Besides honorary degrees from Karlsruhe (1925) and Bonn (1943), he was the recipient (1944) of the A. W. Hofmann Plaque from the German Chemical Society.

[1] Pfeiffer, P., J. Chem. Educ., 5, 1090 (1928); E. Berl, *ibid.*, 19, 153 (1942).

*Symposium on Archaeological Chemistry**

INTRODUCTORY REMARKS

EARLE R. CALEY
The Ohio State University, Columbus, Ohio

THIS symposium may be termed unique, as apparently it is the first on the subject of the application of chemistry to archaeology to be held any where at any time. In a sense it is an experiment to determine the degree and extent of the interest in this field among American chemists. If this experiment succeeds, this symposium may well be only the first of a series.

Instead of being confined to a single aspect of the general subject, the papers on the program today deal with a diversity of topics and thus indicate the variety and range of chemical studies that may be included under the term *archaeological chemistry*.

In a narrow sense this term may be interpreted as denoting only those chemical studies that have a direct bearing on archaeology, but for want of a more suitable broader term it probably should include also other similar studies that may have only an indirect relationship to this science. Thus, under the term *archaeological chemistry* may be included studies on the composition of ancient materials without regard to their immediate archaeological significance, investigations on the decay or corrosion of ancient materials or objects, and experiments on methods for the restoration and preservation of ancient objects of art for the purpose of study or exhibition. By reason of the intimate connection that exists between archaeology and the fine arts it seems reasonable to include also chemical studies of the materials and techniques of the fine arts of all ages except the most recent. Even the problem of the detection of forgeries or alterations of objects of art may also be included. To all this should be added, too, the development of special methods of chemical analysis, microscopy, and the like, suitable for the scientific investigation of ancient materials and objects.

Thus it is evident that we have here a broad field for investigation in the middle ground between chemistry, on the one hand, and archaeology and related subjects on the other, a field that has been but little cultivated by chemists generally. Though it is true that a few investigators have conducted a considerable number of significant investigations in this field, these investigations, more often than not have been highly specialized and sporadic, so that many gaps exist about which nothing is known. One of the most fascinating aspects of this field, it seems to me, are the many opportunities that exist for further investigation, and especially the opportunity that exists for its organization into a unified body of new knowledge.

Let us hope that this present symposium will be regarded in later years as a significant forward step in the development of the field of study that we call *archaeological chemistry*.

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EARLY HISTORY AND LITERATURE OF ARCHAEOLOGICAL CHEMISTRY

EARLE R. CALEY
The Ohio State University, Columbus, Ohio

As a systematic science chemistry is young in years, but archaeology is still younger. Before the last quarter of the nineteenth century archaeological excavation was more often pursued in the spirit of treasure hunting rather than in the spirit of a search for knowledge about ancient peoples and civilizations. The excavations at Olympia, which began in 1875, were probably the first to be conducted on what are now considered to be the correct scientific principles, and adequate scientific reports of the results of excavations are rare before this date. Therefore, it may be said that any scientific relationships between chemistry and archaeology could scarcely exist before about 1875. However, long before this date some chemists had become interested in the composition of ancient materials and had obtained, independent of archaeological considerations, information that is still useful today in the field of archaeological chemistry. The purpose of this paper is to indicate the nature, extent, and value of this early information.

As the writer pointed out in a recent paper,[1] Klaproth was a real pioneer in the chemical investigation of antiquities. However, a few contemporaries of Klaproth also investigated the composition of ancient materials, and at least one paper of these workers appeared in print before any of those of Klaproth. Pearson published a long paper on "Observations on some ancient metallic arms and utensils with experiments to determine their composition" in the *Philosophical Transactions of the Royal Society* for 1796, pp. 395–451, about two years before the date of the first paper by Klaproth.[2] In spite of its length and impressive title, this paper gives only qualitative results and is of little value. A paper by Chaptal[3] is of interest as the first publication on the chemical composition of ancient pigments, though here again only qualitative results are given. Thomson[4] published quantitative analyses of a number of silver coins, among which were a few ancient coins. A quantitative analysis of a specimen of Roman mortar was published by D'Arcet[5] which is of interest as the first analysis of a material of this sort. After the appearance of the last paper by Klaproth, Sir Humphry Davy[6] published an extensive and interesting paper on ancient pigments based on the examination of specimens he himself had collected at Rome and Pompeii, and a short time later he published a brief note[7] on some specimens of Roman pigments found in England.

Between the time of the last publications of Klaproth and Sir Humphry Davy and 1850 only about 25 publications appeared on the chemical composition of antiquities, and no one investigator published more than a single paper. However, many of these papers are of interest, some are of value even today, and one of them is of considerable importance historically. With one exception the publications of this period deal with the composition of metals and alloys. A paper published by John Davy,[8] the only brother of Sir Humphry, is of interest as the first attempt to study the processes of corrosion in ancient metals. The composition of ancient bronzes was studied by a few chemists, among them two very famous chemists, Berzelius,[9] and the elder Fresenius.[10] Among the less well-known chemists who investigated ancient bronzes were Elsner[11] and Knapp.[12] Several papers in this period deal with the composition of ancient Greek or Roman coins. One by Feneulle[13] and another by Walchner[14] contain analyses of Roman Imperial silver coins. A curious point is that each of these papers contains just 13 analyses. Erdmann[15] published six analyses of Greek bronze coins made by students working in his laboratory, and two analyses made by students working under the direc-

[1] J. Chem. Educ., **26**, 242 (1949).

[2] However, Klaproth's first paper was read before the Academy of Sciences in Berlin on July 9, 1795, whereas Pearson's paper was not read before the Royal Society until June 9, 1796.

[3] Chaptal, J., "Notice sur quelques couleurs trouvées à Pompeia," *Ann. chim.*, **70**, 22 (1809).

[4] Thomson, T., "Mémoire sur les monnaies d'argent," *Ann. chim.*, **71**, 113 (1809).

[5] D'Arcet, J., "Analyse du ciment d'une mosaique antique trouvée à Rome," *Ann. chim.*, **74**, 313 (1810).

[6] Davy, H., *Phil. Trans.*, **105**, 97 (1815).

[7] Davy, H., "Observations upon the composition of the colours found on the walls of the Roman house discovered at Bignor in Sussex," *Archaeologia*, **18**, 222 (1817).

[8] Davy, J., "Observations on the changes which have taken place in some ancient alloys of copper," *Phil. Trans.*, **116**, 55 (1826).

[9] Berzelius, J., "Undersökning af metallmassen i några fornlemningar," *Ann. Nord. Oldkundighed*, **1836–37**, 104.

[10] Fresenius, R., "Chemische Analyse einer Celtischen Waffe," *Ann.*, **53**, 136 (1845).

[11] Elsner, L., "Ueber die chemische Zusammensetzung eines chinesischen Metallspiegels," *J. prakt. Chem.*, **34**, 443 (1845).

[12] Knapp, F., "Untersuchung einer antiken Bronze," *Ann.*, **58**, 104 (1846).

[13] Feneulle, H., "Analyse des monnaies d'argent romaines trouvées à Famars," *Ann. chim. phys.*, [2] **32**, 320 (1826).

[14] Walchner, F., "Vermischte Beiträge. I. Untersuchung einiger römischer Münzen, die im Jahre 1825 auf dem Quettich zu Baden-Baden gefunden wurden," *Jahrb. Chem. Phys.*, **21**, 204 (1827).

[15] Erdmann, O. L., "Zusammensetzung einiger altgriechischen Bronzemünzen," *J. prakt. Chem.*, **40**, 371 (1847).

tion of Marchand. However, the most important and extensive series of analyses of coins and other ancient objects composed of copper and alloys of copper was made by Göbel, Professor of Chemistry at the University of Dorpat, who analyzed a total of 54 such ancient objects, 19 of them coins. His results were published in the form of a now scarce pamphlet[16] in which he also lists most of the analyses of his predecessors. Not only is this pamphlet of Göbel important because it contains more original analyses than any single prior work, but it is still more important from the standpoint of the historical development of archaeological chemistry because in it he suggests explicitly for the first time that chemistry may be of service to archaeology and prehistory. Göbel was particularly interested in the source and significance of numerous brass objects excavated in the Russian Baltic provinces. On comparing the composition of these objects with that of European prehistoric bronze objects and with that of Greek and Roman objects of copper and alloys of copper he concluded that they were probably of Roman origin, or at least that they indicated some contact with Roman knowledge of metal working. As a generalization he suggested that ancient copper alloys containing zinc were first produced in the Roman Empire, and that the occurrence of brass objects of like date elsewhere was an indication of contact with Rome by trade, by the utilization of the services of Roman metal workers, or by the transmission of Roman knowledge concerning the production of brass. His conclusions appear to be still valid today. From the standpoint of the history of archaeological chemistry this 38-page pamphlet by Göbel may be considered a classic.

The papers and other works which were published in the period from 1850 to 1875 exceed both in number and importance all those published prior to 1850. Several of the publications of this period are more voluminous than any that appeared before this time, and many contain information that is still very useful today. Though most of the investigators of the period produced only a single paper or other work in this field, a few published two, and one published a long series of papers. As in the period between 1817 and 1850, attention was paid mainly to the composition of ancient metals and alloys, especially bronzes.

In 1850, Donovan[17] published a brief paper on the composition of two prehistoric Irish bronzes, and Salvetat[18] reported analyses of two bronzes of the Roman period found in France. Two papers on the composition of prehistoric Scandinavian bronzes were published in 1852 by Berlin,[19] one a paper containing a series of original analyses and the other a review paper. In the same year the results of a somewhat more general investigation on ancient metals and alloys were published by Phillips,[20] who analyzed a miscellaneous group of 29 Greek and Roman coins, assayed 12 other coins for their silver content, and analyzed eight prehistoric British bronzes. In the following year Mallet[21] published the results of an extensive and important investigation on the composition of prehistoric Irish antiquities. His memoir includes 16 quantitative analyses of bronzes, eight of gold objects, and eight of silver objects. Results of qualitative tests on iron objects, and on amber, jet, and glass beads are also given, and in addition identifications of pigments of medieval date. At about the same time, Giradin[22] published the results of his examination of a great variety of ancient materials and objects. Much of his work was only qualitative and included the examination of gold, silver, and lead objects, glasses, pottery, pigments, colored mortars, and a Roman mural painting. However, he made quantitative analyses of bronzes, coins, and human bones. In view of the evident opportunities which he had for doing much valuable work, it seems unfortunate that most of it was somewhat superficial and that little interpretation of its possible archaeological significance is given. In the year 1853 was also published Layard's "Discoveries in the Ruins of Nineveh and Babylon," an account of archaeological explorations undertaken for the British Museum, which is of considerable interest as the first book on archaeological discovery that has an appendix describing the results of the chemical examination of objects recovered by an expedition. Appendix III of this book contains the first quantitative analysis of Assyrian bronzes, and some comments on the composition of Assyrian glass. Not until the publication of Schliemann's "Mycenae" in 1878 did another such appendix appear, and the practice of publishing in this way results of the chemical examination of excavation finds only became common at a still later date. The chemical reports in the appendices of both these books were written by the famous metallurgist, Percy, though he did none of the actual analytical work. In 1853 also appeared the first of two important papers by Wocel.[23] Though his second paper did not appear until two years

[16] GÖBEL, F., "Ueber den Einfluss der Chemie auf die Ermittelung der Völker der Vorzeit oder Resultate der chemischen Untersuchung metallischer Alterthümer insbesondere der in den Ostseegouvernements vorkommenden, Behufs der Ermittelung der Völker, von welchen sie abstammen," Erlangen, 1842.

[17] DONOVAN, M., "Notice of the analysis of certain gold-coloured bronze antiquities found at Dowris, near Parsonstown in the King's County," *Chem. Gaz.*, **8**, 176 (1850).

[18] SALVETAT, L., "Analyse de deux bronzes antiques de l'époque gallo-romaine," *Ann. chim. phys.*, [3] **30**, 361 (1850).

[19] BERLIN, N., "Om några nordiska metall-legeringars sammansättning," *Ann. Nordisk Oldkyndighed og Histoire*, **1852**, 249; "Några materialier för bedömandet af sammanhanget mellan de antika bronsernas sammansättning och ålder," *ibid.*, **1852**, 254.

[20] PHILLIPS, J., "A chemical examination of the metals and alloys known to the ancients," *J. Chem. Soc.*, **4**, 252 (1852).

[21] MALLET, J., "Report on the chemical examination of antiquities from the Museum of the Royal Irish Academy," *Trans. Roy. Irish Acad.*, **22**, 313 (1853).

[22] GIRADIN, J., "Analyse sehr alter Kunstprodukte," *J. prakt. Chem.*, **60**, 89 (1853).

[23] WOCEL, J., "Archäologische Parallelen," *Sitzungsberichte der Kaiserlichen Akademie der Wissenschaften (Wien), Philosophisch-Historische Classe*, **11**, 716 (1853); **16**, 169 (1855).

later, it is really a continuation of the first, the two together giving the results of the same general investigation. In these papers Wocel not only reports some original analyses of ancient bronzes and summarizes critically many previous analyses, but he also studies the possibility of correlating the chemical composition of bronzes with the place and time of their origin. He shows that such correlation is possible to a considerable extent. His work is a valuable contribution to what should be one of the most important and useful branches of archaeological chemistry. Only a few papers, none of them important, appeared between 1855 and 1860.

Starting with 1860, Fellenberg began the publication of a dozen papers largely devoted to original analyses of prehistoric bronzes. All these papers are scattered through the volumes of the *Mitteilungen der naturforschenden Gesellschaft in Bern* for the years 1860 to 1866, inclusive. His 200 analyses of prehistoric Central European bronzes still constitute the most extensive series of chemical analyses of such bronzes ever made by a single chemist. In the period from 1860 to 1875 a few other investigators, such as Church[24] and Stolba,[25] also published papers on the composition of ancient bronzes and other copper alloys, but the most extensive and important work was done by Bibra, whose book, "Die Bronzen und Kupferlegierungen der alten und ältesten Völker mit Rücksichtnahme auf jene der Neuzeit," published at Erlangen in 1869, is still the greatest single source of information on the composition of ancient copper alloys. Bibra made more chemical analyses of ancient copper alloys than any investigator before or since, and his specimens ranged over nearly all periods and general regions. He also made many analyses of objects of later date. In his book these many analyses are grouped into tables according to localities and periods with some classification according to type of object. His tables also include nearly all the analyses made prior to his time. He not only describes the analytical methods he used but also those of his predecessors. His own analytical methods did not permit very accurate determination of certain components, and his practice of determining copper by difference probably hides many errors of summation. In his interpretations of the analytical results he also gives the views of his predecessors as well as his own. Because of the presence of many misprints and the often indefinite references to the prior literature his book must be used with caution, but in spite of this it is one of the greatest single contributions made to archaeological chemistry by any investigator at any time.

In the same period some attention was also given to the composition of other ancient metals and alloys, and here again Bibra did the most important and extensive work. All his results are contained in a pamphlet entitled, "Ueber alte Eisen-und Silber-Funde," which was published at Nürnberg and Leipzig in 1873. The first part of this important and now scarce pamphlet deals with the composition of iron and steel objects. Out of a total of 60 original analyses 40 are of ancient specimens, the remainder being of medieval and later date. This is the first extensive investigation on the composition of ancient iron and steel. The other part of the pamphlet deals with the composition of ancient silver objects, and contains nearly 100 original analyses of ancient specimens and over 50 of specimens of later date. This pamphlet is still one of the most important single sources of information on the composition of ancient iron and silver objects.

In the period from 1860 to 1875 only a few papers were published on the composition of nonmetallic antiquities. A rather extensive investigation was made by Damour[26] on the density and chemical composition of prehistoric stone objects and of rocks and stones from particular localities with the end in view of providing fundamental data for use in the determination of the point of origin of such objects. No similar investigation appears to have been made since his time. A few papers on the composition of ancient glasses appeared, for example those of Bontemps[27] and of Benrath.[28] A paper by Diamond[29] is of interest as including a mention of the results of an examination by Faraday of a sample of Roman pottery glaze. This famous scientist reported the presence of lead in the sample, apparently the first indication on chemical grounds of the use of lead glaze in ancient times. Finally, a paper by Fontenay[30] gives the first quantitative analysis of a sample of Egyptian blue and describes the first experiments on the synthesis of this famous pigment.

In addition to the publications specifically cited in this paper, there are many others, most of them brief notes, some of them of little importance. Nearly 100 publications prior to 1875 have been located, and possibly a more exhaustive search of the literature would bring the total to well over this number.

This brief survey shows that a considerable amount of work in the field of what we now call archaeological chemistry was done before 1875. Though much of it is only of historical interest, some of it is of considerable practical importance as providing data still useful today. Furthermore, some of this early work includes theories and suggestions for further work that are of no little value. It seems proper to conclude, therefore, that no present worker in this field can afford to ignore the publications of this early period.

[24] CHURCH, A. H., "Analyses of some bronzes found in Great Britain," *J. Chem. Soc.*, **18**, 215 (1865).

[25] STOLBA, F., "Analyse alterthümlicher Bronzeobjecte aus der Sammlung des böhmischen Museums," *J. prakt Chem.*, **101**, 139 (1867).

[26] DAMOUR, A., "Sur la composition des haches en pierre trouvées dans les monuments celtiques et chez les tribus sauvages," *Compt. rend.*, **61**, 313, 357 (1864); **63**, 1038 (1856).

[27] BONTEMPS, G., "Examen des vitres de Pompei," *Compt. rend.*, **54**, 980 (1862).

[28] BENRATH, H., "Beiträge zur Archäologie weissen Glases," *Wagner's Jahres-Bericht der Chemischen Technologie*, **15**, 340 (1869).

[29] DIAMOND, H., "Account of wells or pits, containing Roman remains, discovered at Ewell in Surrey," *Archaeologia*, **32**, 451 (1867).

[30] FONTENAY, H. DE., "Note sur le bleu égyptien," *Ann. chim. phys.*, [5] **2**, 193 (1874).

THE CORROSION PRODUCTS OF AN ANCIENT CHINESE BRONZE

RUTHERFORD J. GETTENS
Fogg Museum of Art, Harvard University,
Cambridge, Massachusetts

THE corrosion of metals is not only a serious industrial problem and a household nuisance but it is also an interesting chemical phenomenon. It is often a complicated process which produces several distinct products in intimate juxtaposition. Some of the best examples of the complexity of this process are seen occasionally in the patina of bronze vessels and utensils excavated from the early graves of China. The diversity of copper and tin compounds disposed in the layered structures on their surfaces testify to the numerous factors that must enter into the formation of the mineralized envelope. To the collector these structures and products are of interest because their color and texture frequently enhance the value and reflect the age and authenticity of a piece; chemists and metallurgists might well study old bronze objects because the slowness of formation of the corrosion layers and the segregated distribution of the mineral products undoubtedly could throw more light on the corrosion mechanism.

Most of the observations reported here are not new. Beginning with Berthelot (*2*) several investigators, including Rosenberg, (*12*), Fink and Polushkin (*5*), Collins (*4*), Caley (*3*), Plenderleith (*11*), and the author (*7, 8*) have described the layered structure on the surface of ancient corroded bronzes and have suggested chemical and electrochemical reactions and mechanisms to explain the formation of the several mineral products contained in them. Only a few Western investigators, however, have had opportunity to study the corroded high-tin cast bronzes from the Far East, principally, I think, because there has been so little study material available. Collectors have brought from China mainly whole and intact pieces of museum quality and have left the scraps and broken fragments behind. It was our good fortune sometime ago to be given a portion of an ancient bronze ceremonial vessel of the Chou period (112–255 B.C.) with freedom to do as we pleased with it. This vessel, with wide oval mouth and bulbous body, called a *hu* was purchased as a whole object in China during the war. Nothing was known of the exact origin of the bronze or the circumstances of its burial. It was shipped to this country but on arrival was found broken in several pieces (Figure 1). Examination showed it was, in part, a forgery. An incomplete lot of broken fragments of a bronze *hu* was found, probably in a burial; to complete the object and to make it saleable some Chinese artisan cut out and shaped pieces of heavy plate copper to fill in the voids. The old fragments and new replacements were joined with soft solder to make a complete shape, and finally the new metal surfaces and joins were cleverly concealed with an artificial patina made from green-colored stucco. The solder had adhered poorly to the old corroded metal, and the object fell apart in transit. In its broken condition there was no difficulty in telling the old from the new parts. While the trumped-up parts were amusing, the original earth-corroded pieces were of chief interest because of their value for analytical purposes.

Figure 1

This fragment of a Chinese bronze vessel of the first millenium B.C. is covered on the left side with the natural corrosion products of copper and tin. The deeply modeled features on the right are done in plaster and are artificially colored. The faking was done to make a broken object appear to be intact.

The bronze was cast, probably by the well-known *cire perdu* method. The surface was modeled in low relief with conventionalized animal forms set against delicate scroll and maze patterns so commonly used in that period. The surface in places is smooth gray-green in color but interrupted with irregular patches of red and bright and dull green. The patina is hard and adherent. A few small fragments were mounted in bakelite and polished for metallographic study. Microscopic examination revealed that there are three principal zones: (1) in places, in the center, small irregular areas of uncorroded metal, (2) an intermediate partially mineralized zone, and (3) an outer completely mineralized zone. The outer and intermediate zones have a layered structure within themselves (Figure 2).

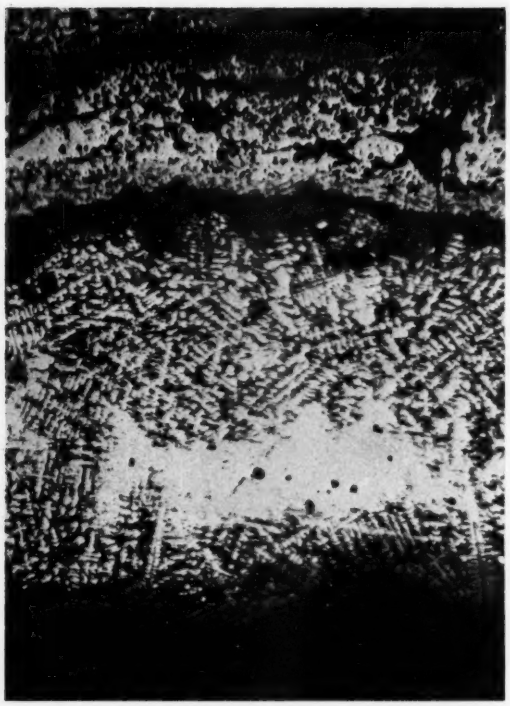

Figure 2

This unetched polished cross section of the old metal shows the three principal zones: (1) uncorroded metal core, (2) intermediate partially mineralized zone, and (3) outer completely mineralized zone. The dark gray band just outside the zone of dendrites is cuprous chloride. The bright reflecting porous layer beyond that is red cuprite. (Bright field, 50×)

It was necessary to know the composition of the original metal. Although corroded completely through in places there are parts where the unchanged metal is still 1 to 2 mm. thick. Samples from these areas were cut out, carefully cleaned to remove oxidation products, and analyzed by standard methods. The results seen in the table show that it is a high-tin (above 20 per cent) bell metal, and it is just short, in respect to tin content, of being speculum metal, the alloy that was so extensively used in China at a little later period for casting mirrors. Occurrence of lead in this type of bronze is not unusual and this amount of 4 to 5 per cent is somewhat more lead than such a copper-tin alloy can take into solid solution. Some unalloyed lead is dispersed in tiny globules throughout the alloy matrix. Spectrographic analysis showed the presence of several trace elements but among them arsenic was the only one reported in the order 0.1 to 1.0 per cent.

The corrosion zones and the several mineral layers were of chief interest. Most striking is the intermediate penetration zone where the eutectic (tin-rich phase) has been converted to a black product leaving dendrites of alpha bronze (copper-rich phase) unattacked. It is a natural etch. In the casting this bronze had apparently cooled slowly, allowing alpha dendrites to form freely and nearly perfectly (Figure 3). This zone of interphase penetration is generally 1 to 3 mm. thick, but in places it goes entirely through the metal. Because it is so intimately mixed with uncorroded alpha it was quite impossible to isolate the black material for microchemical tests. It was possible, however, from a polished section, to isolate chips free from outer corrosion product and to subject them to X-ray diffraction analysis. The powder pattern showed mainly an expanded copper lattice, but in addition it showed faint lines characteristic of cassiterite and also one unidentified line. No lines of the copper chlorides were observed. Tin oxide could be expected, and, indeed, white tin oxide is plentifully seen in the outer corrosion layers. The faintness of the cassiterite lines indicate that the tin oxide is in a cryptocrystalline state. Why does it seem to be black here (Figure 4)? It is possible that it is stained black with cupric oxide, and it is well known that some forms of cupric oxide (like melachonite) are so nearly amorphous that they give indistinct diffraction patterns or none at all. If the black is cupric oxide it is doubtful if it is formed by direct oxidation of copper especially under the reducing conditions that seem to prevail in the interior after corrosion attack is well along. There is a remote possibility that the black is formed from cupric hydroxide precipitated at cathodic areas of local cells set up by electrolytic action in the salt environment. J. W. Mellor (*10*) cites (Vol.

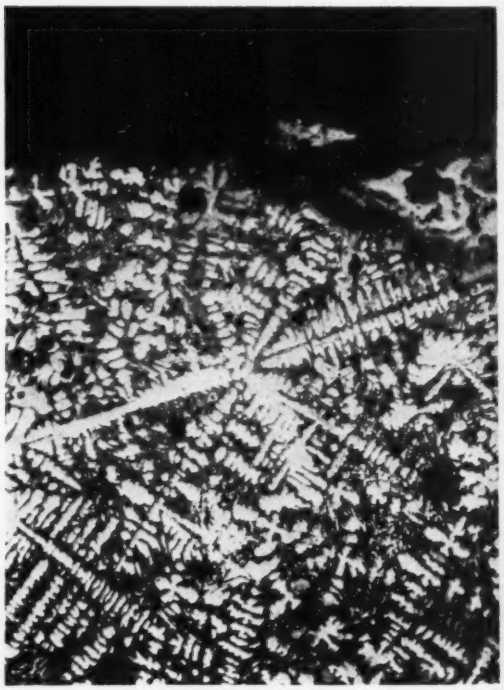

Figure 3

The original duplex structure of the cast copper-tin alloy is revealed by the corrosion and darkening of the high-tin phase which surrounds the crystals of copper-rich alpha bronze. (Bright field, 100×)

III, p. 144) evidence that cupric hydroxide hydrogel in contact with water or salt solution can eventually dehydrate and turn black even below room temperatures although several months may be required to bring about the change. Bengough and May (*1*) state (p. 103) that in copper corrosion dark brown or black hydrated cupric oxides are important constituents of scales that form on the copper surface. This explanation of the initial black corrosion product is, however, highly conjectural since we have so little direct analytical information bearing on it.

On the outer edge of the intermediate penetration zone the alpha dendrites are attacked, and only disconnected remnants or islands of metal are left. From here outward only mineral corrosion products are found. The first of these is a grayish soft waxy layer (Figure 2) which was recognized as cuprous chloride (nantokite). This is the key mineral product and indicates that salt in soil water was perhaps the main corroding factor. It is not at all unusual to find cuprous chloride in ancient corroded bronzes from desert areas or other places where salt is present. It is the material on old bronzes which gives rise in museums to the phenomenon called "bronze disease" which is nothing more than the oxidation and hydrolysis in humid weather of cuprous chloride to basic cupric chloride. When a polished cross section of the metal was exposed for a few hours in a humid chamber a narrow line of bright green blistery eruptions formed just along the zone of gray cuprous chloride (Figure 5). When a sample of the pure translucent waxy material was dug out and treated with a drop of dilute hydrochloric acid and rapidly dried it was observed that the residue was a mixture of pale green crystals of cupric chloride and colorless tetrahedral crystals of cuprous chloride.

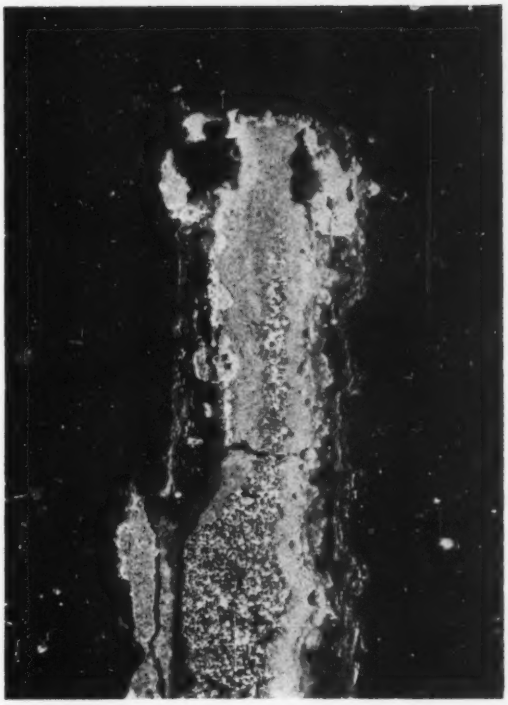

Figure 5

This is a freshly polished cross section of the corroded metal after it was placed in a humid chamber for some hours. On either side of the metal core bright green blisters and eruptions of basic cupric chloride caused by oxidation and hydration of the cuprous chloride inner layer have formed. (Dark field, 9×)

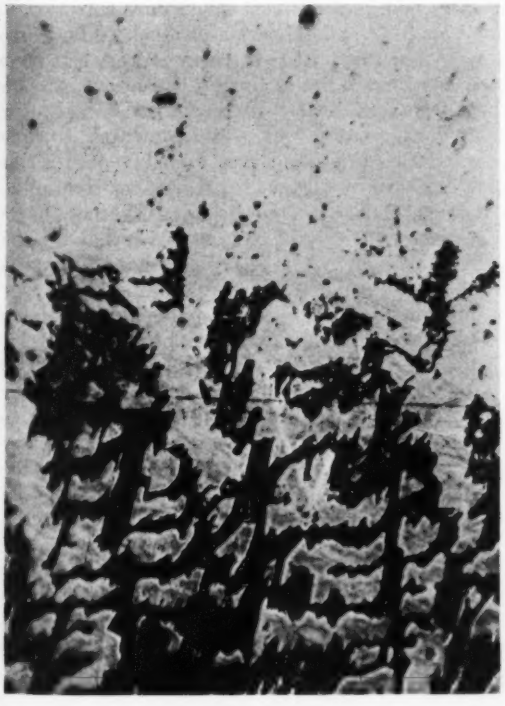

Figure 4

The inner advance front of corrosion as it penetrates among crystals of alpha bronze is here clearly seen. The initial corrosion product is dark in color. (Bright field, 425×)

Going outward, next comes a narrow zone of redeposited metallic copper. This is compact but crystalline as in an electroplate (Figure 6). Strangely it is not continuous but intermittent. Redeposited copper occurs also in tiny pockets throughout the initial penetration zone among dendrites of residual alpha. Cathodically-formed copper is often observed also in corroded modern brass pipes and fittings. When formed, either in the dezincification of brass or in the destannification of bronze, redeposited copper indicates the electrochemical nature of this kind of corrosion.

Next comes a thick band of cuprous oxide (cuprite) which is bright red in dark field illumination and blue-gray in bright field. In places where there are small voids in the cuprite crust the corners of nearly perfect cubes of that mineral jut into the hollows (Figure 7). It is well known that in presence of water and air cuprous chloride is easily converted to cuprous oxide as

well as to basic cupric salt. The density and compactness of the cuprite layer is probably an important factor in preventing the complete oxidation of cuprous chloride. It also explains the difficulty often encountered in attempts to rid the bronze of unstable cuprous chloride by electrolytic reduction or by other means.

Tin oxide seems to form no separate distinct interior layer. At high magnifications, however, white tin oxide is seen deposited mainly in little disconnected seams throughout the cuprite, like streaks of fat in a beefsteak. Ghosts of the original dendritic structure in places still persist in the white tin oxide. The tin corrosion product, unlike the copper, does not migrate because after formation it immediately becomes so insoluble. The initial products of copper corrosion, which are slightly soluble in the corrosion liquid, either deposit cuprite around the tin oxide or they are leached out leaving a nearly solid tin oxide patina, as is often seen on ancient Chinese bronze mirrors (*9*). In certain areas on the surface copper is nearly all dissolved out leaving a smooth solid tin oxide. It would seem that the copper in a bronze could be dissolved out completely

Figure 6

The narrow light band above and the broad one below are of redeposited copper. One small pocket of redeposited copper can also be seen among the residual dendrites in the partially mineralized zone. (Bright field, 50×)

leaving a fragile skeleton of tin oxide as the only indication of the original shape.

The predominent exterior corrosion product in other areas is green crystalline basic cupric carbonate (malachite) mixed with some basic cupric chloride. This basic chloride, which an X-ray powder pattern showed to be paratacamite, occurs mainly in little pockets and patches of bright green powdery material. Frondel (*6*) has recently showed that paratacamite, dimorphous with atacamite, is found among the corrosion products of ancient copper and copper alloys. The considerable amount of malachite present supports the observation

Analysis of a Chinese Bronze Ceremonial Vessel

	Sample from rim (average of duplicate analyses)	*Sample from base (average of duplicate analyses)*
Copper	73.8%	72.3%
Tin	20.3%	21.8%
Lead	4.6%	4.1%
Iron	0.0%	...
Zinc	Trace	...
Total	98.7%	98.2%

of Fink and Polushkin that the ultimate product of copper corrosion is the basic carbonate, stable in the presence of carbon dioxide.

No corrosion products of lead were isolated or identified. Lead, however, may play a more important role in the corrosion process than we realize especially if it is dispersed as a separate metal phase in the duplex alloy structure.

As these observations bear out, bronze corrosion is a complex process, and there is still much to be learned about it. I doubt if the corrosion process can be expressed in a neat series of chemical reactions, one proceeding from the other. I think Fink was correct when he suggested: "The fundamental corrosion reactions, while important, are probably only a small part of the story. Physical retention of semi-plastic corrosion products in an envelope about the metallic core may play a greater part in layer formation than the original corrosion reactions." The solid metal crystal structure appears to have been invaded by an ion-bearing solution, but how the ions migrated within this solid structure is hard to explain. I picture, however, the existence, even in the interior, of microscopic colloidal pipes like those seen during the precipitation of copper ferrocyanide and such as exists in a colloidal garden. Juxtaposed anodic and cathodic areas and tiny concentration cells no doubt are involved. Blisters and locally corroded areas on the surface are evidence of this. The products we now see are probably, in part, solidified colloidal gels formed after the bronze was taken from the wet earth and dried. Existence of dried colloidal gels may be the explanation for the diffuse X-ray diffraction lines produced by the inner corrosion products.

Although I may have seemed to disparage the employment of chemical step reactions to explain such a complex process, yet the layered structure does indicate they are to an extent valid and there is independent experimental evidence to explain the mode of formation of some of the end products. Space does not permit a

complete discussion here, but Caley's writings may be referred to for more extensive treatment of the theoretical considerations. To explain the observations made on this particular object, however, the following chemical reactions drawn from many sources might be used:

In the inner penetration zone:

At anodic areas:

$$\underset{\text{(bronze)}}{\text{Cu-Sn}} \rightarrow \text{Cu}^{++} + \text{Sn}^{++}$$

$$\text{Cu} + 2\text{NaCl} + \text{H}_2\text{O} + [\text{O}] \rightarrow \underset{\text{(anodic)}}{\text{CuCl}_2} + \underset{\text{(cathodic)}}{2\text{NaOH}}$$

$$\text{CuCl}_2 + 2\text{NaOH} \rightarrow \underset{\text{(blue gelatinous)}}{\text{Cu(OH)}_2} + 2\text{NaCl}$$

$$\text{Cu(OH)}_2 \rightarrow \underset{\text{(black)}}{\text{CuO}} + \text{H}_2\text{O}$$

Simultaneously:

$$2\text{Sn} + \text{O}_2 + 2\text{H}_2\text{O} \rightarrow 2\text{Sn}^{++} + 4\text{OH}^-$$

$$\text{Sn}^{++} + 4\text{NaCl} + 2\text{H}_2\text{O} + 2[\text{O}] \rightarrow \text{SnCl}_4 + 4\text{NaOH}$$

$$\text{SnCl}_4 + 4\text{NaOH} \rightarrow \underset{\text{(white gelatinous)}}{\text{Sn(OH)}_4} + 4\text{NaCl}$$

In the outer mineralized zone:

Copper from unattacked alpha phase reduces CuCl_2

$$\text{Cu} + \text{CuCl}_2 \rightarrow \underset{\text{(nantokite)}}{2\text{CuCl}}$$

In water and air:

$$4\text{CuCl} + [\text{O}] \rightarrow \underset{\text{(cuprite)}}{\text{Cu}_2\text{O}} + 2\text{CuCl}_2$$

In excess NaCl:

$$6\text{CuCl} + 3[\text{O}] + 4\text{H}_2\text{O} \rightarrow \underset{\text{(atacamite)}}{\text{CuCl}_2 \cdot 3\text{Cu(OH)}_2 \cdot \text{H}_2\text{O}} + 2\text{CuCl}_2$$

In presence of carbon dioxide:

$$\underset{\text{(malachite)}}{\text{CuCl}_2 \cdot 3\text{Cu(OH)}_2 \cdot \text{H}_2\text{O}} + \text{CO}_2 \rightarrow \text{CuCO}_3 \cdot 3\text{Cu(OH)}_2 \cdot 2\text{H}_2\text{O} + 2\text{HCl}$$

Simultaneously:

$$\text{Sn(OH)}_4 \rightarrow \underset{\text{(cassiterite)}}{\text{SnO}_2} + 2\text{H}_2\text{O}$$

Cupric ions in contact with Cu-Sn eutectic

$$\text{Cu}^{++} + \text{Sn} \rightarrow \text{Sn}^{++} + \underset{\text{(redeposited copper)}}{\text{Cu}}$$

ACKNOWLEDGMENTS

The fragments of the old bronze on which this work was done were loaned by Mr. Charles Fabens Kelly, Curator of Oriental Art of the Chicago Art Institute. The author is greatly indebted to Mr. Daniel Cushing, consulting metallurgist of Boston for the metallographs used to illustrate the paper; Mr. Cushing has also taken some very fine color transparencies of these subjects which unfortunately cannot be reproduced here. Special acknowledgment is also made to Professor Clifford Frondel and to Miss Mary Mrose of the Dana Laboratory, Department of Mineralogy, Harvard for the X-ray diffractions analyses and to Professor H. C. Harrison of Rhode Island State College for the spectrographic analysis. Mr. Charles Fletcher of the Department of Geology mounted and polished the specimens for metallographic study. Mr. Earl Robertson, now of Eastman Kodak Laboratories, made the chemical analyses.

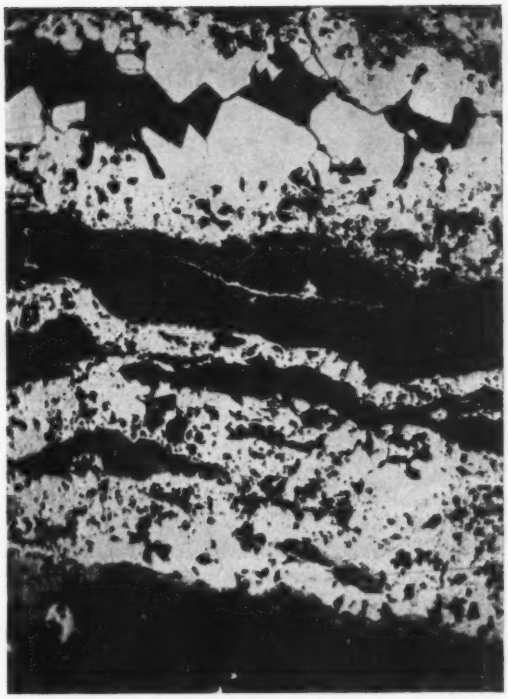

Figure 7

The red cuprite zone is banded and fissured. The right-angled corners of nearly perfect cubes of cuprite project into the wide fissure seen near the top of the photomicrograph. (Bright field, 100×)

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ANCIENT PIGMENTS

Particularly Second Century B.C. Pigments from Corinth

MARIE FARNSWORTH

Metal and Thermit Corporation, Rahway, New Jersey

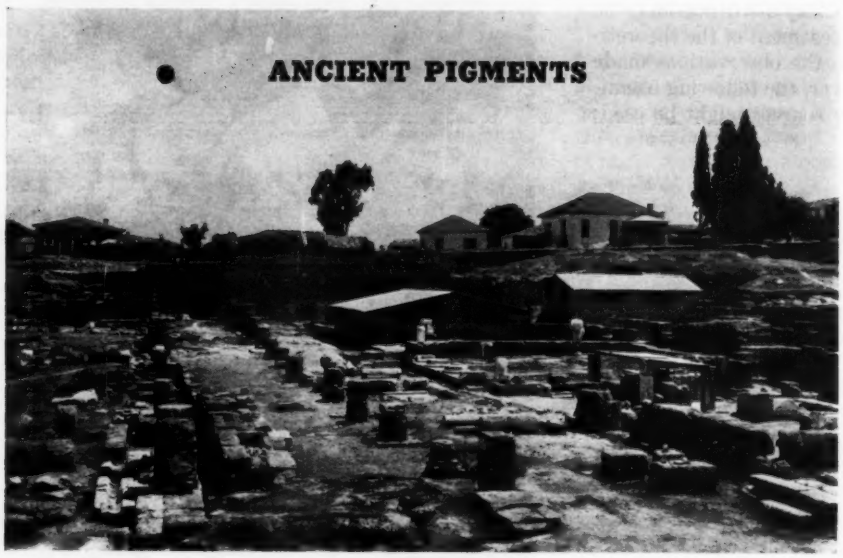

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Figure 1. Corinth: East End of South Stoa, with Walls of the Roman Period, from West

Ancient pigments have always aroused considerable interest, not only for archaeologists but also for art historians and for chemists. The first two papers on the subject written by chemists appeared at the beginning of the nineteenth century. They were written by the well-known British chemist, Sir Humphry Davy,[1] and by the Frenchman, M. Chaptal.[2] These papers are fascinating reading today for anyone interested in ancient pigments, not only for the variety of Roman pigments uncovered but also for the ingenious methods used on small quantities of material in the days before modern methods of analysis were available. Since then pigments have been recorded during the course of many excavations in Egypt, Greece, and Italy, and the ancient palette is fairly well known to us. For information we have lumps of pigments found in excavations, wall paintings, architectural decorations, terracotta decorations, literary sources, etc. The most desirable from the standpoint of the chemist are lumps of pigment, for then enough material is available for whatever tests are needed for complete identification. In no case, however, is it desirable to destroy the entire sample for experimental purposes, for a part should always remain at the excavation site with the other objects found there.

Because of the large amount of material available, the unequivocal dating, and the presence of at least one unusual pigment, a group of samples recently sent to me from Corinth, Greece, by Professor Oscar Broneer, is of considerable interest. Regarding these pigments Dr. Roger Edwards, who worked with Professor Broneer at Corinth, wrote, in a letter, as follows:

A brief account of the well from which the pigments come has been published in *Hesperia*, XVIII (1949), p. 152.

The well is one of a connected series of thirty-one which supplied water to the shops of the South Stoa at Corinth and is located in Shop XIX (numbering from the east). The series was constructed in direct relation to the Stoa and was an integral part of the building. Present opinion favors a date in the second half of the fourth century B.C. for the construction of the Stoa: the well series was probably made soon after its completion. A general account of the wells is given in *Hesperia*, XVI (1947), pp. 239 ff.

In this particular well there were four distinct fillings, three of which bear relation to the date of the pigments. The first, from −10.00 m. to the bottom, −10.70 m., contained the material which accumulated during the period of use of the well as a water supply, and is dated in the third century B.C. The second, from −8.00 m. to −9.20 m., separated from the first by a layer of sherds and pebbles covered with an adhesive white substance, was an accumulative filling which gathered after the well had ceased to be used to supply water; according to its pottery and coins it should be dated in the first half of the second century B.C. The third filling, from which the pigments come, filled the remainder of the well nearly to the top, and consisted of a mass of destruction debris to be associated with the destruction of Corinth by Mummius in 146 B.C. This material included a considerable quantity of broken roof-tiles and architectural fragments derived from the Stoa itself, as well as great quantities of pottery and pigments and numerous other objects of various types. This filling was thrown in the well in a single operation, presumably in a clean-up of the area soon after the establishment of the Roman colony of Corinth in 44 B.C., although there was no admixture of Roman objects in it. With the filling were associated a large number of coins of which those most pertinent for the dating are a group of 34 Ptolemaic coins of bronze which Miss Margaret Thompson is publishing separately. Her opinion, expressed in a preliminary draft of her article which she has sent me, is that 32 of these coins are to be assigned to Cleopatra I during the period of her regency (*ca.* 180 to 175 B.C.), and the remaining

[1] DAVY, HUMPHRY, "Some experiments and observations on the colours used in painting by the ancients," *Phil. Trans.*, **105**, 97–124 (1815).

[2] CHAPTAL, M., "Sur quelques couleurs trouvées à Pompeïa," *Ann. Chim.*, **70**, 22–30 (1809).

two to Ptolemy VI and VIII during their joint reign, 170–164 B.C. She considers the group a hoard accumulated between *ca.* 170 and 168 B.C. The dating for these coins, the character of the filling, and the presence of a filling beneath dating in the first half of the second century B.C. make the assignment of the filling to the destruction of Mummius in 146 B.C. virtually certain.

Because of the presence in the filling of architectural pieces derived from the South Stoa it would seem most likely that many, if not necessarily all, of the other objects were originally part of the equipment of the shop in which the well was located, and that this material was likewise involved in the destruction of 146 B.C.; *i. e.*, that it was in use or on hand at that time. The impressive quantity and variety of pigments in this filling might tend to suggest that a factory for the manufacture of terracotta figurines, architectural terracottas, pottery, or the like was located here; but no significant amount of objects to suggest this identification of the source of the pigments as an establishment of that kind was found with them. The Stoa shops, in fact, were extremely small and not adapted at all to such undertakings. Alternatively, it seems most likely that we have to do here with a supply shop. Some of the pottery in the filling was certainly used in connection with the pigments, for a number are stained on the interior with color; one, a foreign import to Corinth (*Hesperia*, XVIII (1949), pl. 16, No. 15), bears a dipinto inscription referring to cinnabar, from which the color vermilion was obtained. Mixed with the filling were amazing quantities of bronze nails and tacks, many unused, as well as iron spikes. These, coupled with the extensive range and quantity of the pigments and other substances tend to suggest the modern hardware store where paints, nails, and associated materials for house construction and decoration are on sale. This shop may well have been an establishment of that type on a small scale.

Figure 1 shows the area at Corinth where the South Stoa was excavated.

In establishing the nature of pigments and related materials it is usually advantageous to examine them spectrographically to find the principal element or elements and then proceed to chemical, microscopic, and X-ray tests as indicated. By following this general procedure five of the samples sent were found to be ochres. An ochre is a natural earth which owes its color to iron oxide, either hydrated or anhydrous. Red ochre (miltos) is colored by anhydrous iron oxide, Fe_2O_3, but in brown and yellow ochres the color is caused by various hydrated forms of iron oxide. Of the five ochres examined, two were definitely red ochres and were very similar. One was definitely yellow ochre. The last two were yellowish but might better be classified as brown. One of these contained a considerable amount of calcium carbonate which appeared to have been found in nature with the earth and not to have been added intentionally.

In addition to the samples of ochre there was a buff sample which was found to be clay and a reddish orange pigment which was the mineral realgar, AsS. There was also a sample of wood charcoal, but it seems unlikely that it was meant for a pigment for it still contained a considerable amount of tarry material. Two samples, one white and the other somewhat grayish in tone, were identified as dolomite, $Ca(Mg)(CO_3)_2$. There was also a sample of gypsum, $CaSO_4 \cdot 2H_2O$, which was a stone rather than a pigment, of the type commonly called alabaster. Included was a transparent tabular crystal of the mineral anhydrite, $CaSO_4$. Also sent were two unusually good specimens of red tetragonal litharge with only a thin outer coating of normal lead carbonate. These lead slags, a by-product of silver manufacture, were probably brought from Sunium in ancient times.

Most of the ancient pigments, as is suggested by the above examples, are naturally occurring substances. At most, they were ground in water and floated to obtain finely divided particles. Yellow ochre was burned to produce red. One manufactured pigment that was used extensively and over a long period of time is Egyptian blue or blue frit. This was manufactured by heating a mixture containing silica, a copper compound (probably malachite), calcium carbonate, and natron (a naturally occurring compound of sodium carbonate and sodium bicarbonate). Laurie[3] has investigated the manufacture of this pigment and has shown that it can only be formed in a very narrow temperature range (830–850°C.). In spite of the careful control required, both of the purity of the ingredients and of the temperature of its manufacture, specimens as early as the fourth dynasty (2900–2750 B.C.) have been found. While this pigment was used extensively in Greece there is no evidence that it was ever manufactured there. It was, however, later manufactured in Italy and was the universal blue for fresco painting in Rome in Imperial times. It disappeared from the artist's palette sometime between the second and seventh centuries, according to Laurie, and is not used at the present time.

Permission of the American School of Classical Studies, Athens, Greece.

Figure 2. Jar with Inscription Referring to Cinnabar

Several lumps of this pigment were sent from Corinth, some pure blue and others greenish in tone. The latter color probably resulted from overheating, as suggested by Laurie, and not because of an excess of iron; for spectrographic analysis showed the blue and greenish samples to contain about the same amount of this element.

After I had reported on these samples and the two discussed below, Dr. Edwards wrote that one of the pots (Figure 2) had a painted inscription referring to

[3] LAURIE, A. P., "The Technique of the Great Painters," Carroll and Nicholson, London, 1949, p. 25.

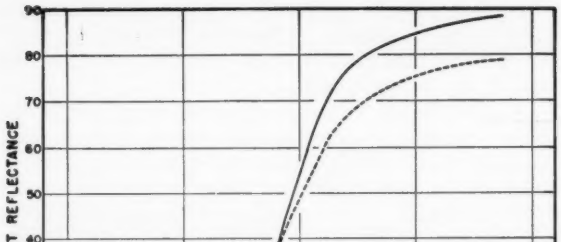


Figure 3. Reflectance Curves for Ancient and Modern Rose Madder

cinnabar and was stained a corresponding color on the interior. Spectrographic analysis of scrapings from this pot showed that it had at one time held a mercury compound although no lump of cinnabar was among the samples sent.

By far the most interesting pigment in this interesting collection was a sample of rose madder. Very little definite information regarding the use of rose madder as a pigment in ancient times occurs in archaeological or chemical literature. Art historians make statements based on insufficient evidence and conflicting opinions are sometimes stated verbally. For these reasons and also since enough material of unequivocal date was available for definite identification, this particular Corinth pigment is of more than passing interest. At first one lump of the rose madder pigment about $^1/_2$ inch in diameter was sent. Later, at my request, three smaller lumps about $^1/_4$ inch in diameter were also sent. I believe there are still three of the smaller lumps at Corinth. The lumps had a thin layer of calcareous dirt on the surface but as soon as that was removed the entire sample had a uniform pink color tending toward the violet. At about the same time that the pigment samples were sent from Corinth, Dorothy Thompson sent from the Agora a very small amount of sample which resembled the rose madder from Corinth but which was more purplish in tone. This sample had been scraped from a scrap of a bowl, P17975, with color adhering to the inside, which was a very small piece of a large dish. Regarding this sherd Dr. Edwards wrote as follows:

> The fragment resembles most closely a plate of the type of Agora Inv., P5984, of which there are several examples on our shelves. It seems to come from an extremely large plate since, though it is difficult to estimate the diameter from so small a fragment, it seems to have been at least 0.45 m. in diameter.
>
> As far as the Agora evidence goes, this type with low rim, straight in profile, seems not to put in an appearance much if at all before the end of the second century B.C. This is corroborated by the evidence of Mummian destruction fills at Corinth (146 B.C.) in which the type does not occur.

Most of the tests mentioned below were of necessity carried out on the larger specimen from Corinth. Enough tests, chemical, spectrographic, and microscopic, were made on the smaller sample from Athens, nevertheless, to leave no doubt as to its constitution. Spectrographic examination showed aluminum to be the principal constituent of both samples. This fact was also confirmed by a chemical test, *i. e.*, a small amount was dissolved in hot 10 percent sodium hydroxide solution and, after acidifying with hydrochloric acid, aluminum hydroxide was precipitated with ammonia. The fact that the material first turned black on heating and then a dirty gray showed the presence of organic matter. These tests, together with appearance, led me to believe that both samples were rose madder.

Because of the interest of the Fogg Museum in pigments I sent both samples to Mr. Rutherford J. Gettens of the Technical Department. He very kindly made a thorough microscopic examination of the specimens and agreed that both appeared to be madder.

Permission of the Metropolitan Museum of Art, New York.

Figure 4. Terracotta Statuette with Rose Madder Coloring

He made the further interesting observation that the Agora sample was actually a mixture of blue frit and rose madder which accounted for the purplish tone. Spectrographic analysis had shown more copper present in this sample than in the Corinth one, which showed only a very small trace, but had not, of course, indicated in what form it occurred. In spite of all these convincing data we both agreed that a specific test for madder was necessary in order to settle beyond question the nature of the pigment.

Madder is a natural dyestuff from the root of the herbaceous perennial, *Rubia tinctorium*, which is native to Greece. Its principal coloring agent is alizarin (modern turkey red) modified by purpurin, another organic dyestuff. In preparing pigments now the root extract is added to alum and precipitated with an alkali. Aluminum hydrate thus acts as a carrying agent for the dye. The making of madder lakes is a difficult process and even today old established color firms have their own recipes which they are unwilling to publish. In spite of these technical difficulties the Greeks, in so far as we have been able to observe, used a method almost identical with the one in use at present.

The dyestuffs in madder have definite chemical properties but what is even more characteristic and more easily observed is their specific absorption or reflectance of visible light. To make an absorption curve it would be necessary to remove the dye from the alumina with alcohol or some other solvent and thus the pigment would be destroyed. A reflectance curve can, however, be made without harming or altering the pigment in any way. Figure 3 shows the reflectance of the Corinth pigment in the visible which was made on a Beckmann spectrophotometer. In the same figure is a similar curve for a modern sample. These curves leave no doubt that we have here a sample of rose madder which, although more than 2000 years old, is practically identical with the modern sample. This characteristic reflectance (*ca.* 510–550 mμ) is, incidentally, from purpurin and not from alizarin. The lesser reflectance of the ancient madder in both the shorter and the longer wave lengths indicates that the coloring matter is not as pure as in the modern sample.

Most archaeologists are acquainted with the color of rose madder, or Hellenistic pink, examples of which are undoubtedly to be found on terracotta statuettes in many museums, although this supposition has not been checked by chemical or physical tests. Figure 4 shows a terracotta statuette from the Metropolitan Museum in New York where the coloring matter is almost certainly madder. Figure 5 shows a similar figurine where the coloring matter is ochre. Actual unequivocal identifications of madder dated anywhere near the samples reported are very few. Two possible examples of rose madder were reported by chemists early in the nineteenth century. Sir Humphry Davy (*op. cit.*) examined a broken vase of earthenware in the baths of Titus which contained a pale rose color fixed on clay. He was unable to decide whether it was of animal or vegetable origin. At about the same time Chaptal (*op. cit.*) reported on some pigments found in a shop at Pompeii. One, rose in color, he reports as a true lake on an alumina base. Although no specific test for madder was made in either case, the identifications as given are probably correct.

The example most often mentioned is the one found at Hawara by Petrie. Lucas[4] mentions it as a pink color from a tomb painting but actually the find

Permission of the Metropolitan Museum of Art, New York.

Figure 5. Terracotta Statuette with Ochre Coloring

consisted of six pots of paint of the Greco-Roman age. These were identified by the chemist Russell[5] by modern chemical means. One, pink in color, he identified as madder on a calcium sulfate (gypsum) base. Since he was able to get the absorption spectrum of purpurin

[4] LUCAS, A., "Ancient Egyptian Materials and Industries," 3rd ed., Edward Arnold and Co., London, 1948, p. 397.

[5] RUSSELL, W. T., "Egyptian Colours," Chap. VIII, pp. 44–48, in W. M. FLINDERS PETRIE, "Medum," London, 1892.

from this pigment there is no doubt as to its identity. These pots of paint are now in the British Museum. Lucas (*op. cit.*) also mentions that there are examples of Roman madder in the Naples Museum.

These four examples are all later than the two reported in this paper and thus the Corinth sample appears to be the earliest to be studied. As a matter of fact, it appears to be the only early example, with the exception of the Egyptian one, that has been definitely identified. Laurie,[6] who has written extensively on pigments has, on the basis of the Egyptian pigment, assumed that the ancients always used a gypsum base. The Corinth pigment has definitely shown, however, that the Greeks prepared madder lakes on alumina, which are very similar to modern madder lakes. This use of alumina is not surprising since alum was well known to the ancients and was used for a mordant in dyeing. It might be mentioned that madder, also, was apparently used extensively for dyeing in ancient times.

[6] LAURIE, A. P., "Ancient pigments and their identification in works of art," *Archaeologia*, **64**, 317 (1913).

I have not gone into this question for I have nothing new to add. For what purposes madder pigment was used is not entirely known but we do have much indirect evidence that one use, at least, was for coloring terracotta statuettes.

In conclusion it might be interesting to list the pigments used by the ancients. Various studies have definitely established the use of the following:

Whites: chalk, gypsum, and white lead.

Blacks: charcoal, lamp and bone black, and, to a certain extent, pyrolusite.

Yellows: yellow ochre and orpiment.

Reds: red ochres, cinnabar, realgar, and later, red lead.

Pink: madder or a mixture of red ochre and white, principally the latter.

Blues: azurite and Egyptian blue.

Browns: ochres.

Greens: malachite, chrysocolla, green frit or usually a mixture of blue frit and yellow ochre, verdigris, and terre verte.

GOLD-COPPER ALLOYS IN ANCIENT AMERICA

WILLIAM C. ROOT
Bowdoin College, Brunswick, Maine

THE first mention of the use of alloys of gold and copper by the Indians of America is found in a Spanish account of an expedition by Grijalva to Yucatan in 1518.[1] The Spaniards asked the Indians if they had any gold. "...They said 'yes' and brought some *guanines* which they put in their ears and some round discs of *guanin* and said they had no other gold than that...so that the reader may understand what *guanines* are...I may say that they are pieces of gilded copper, and if they contain any gold it is very little."

Other examples of this copper-gold alloy were soon found. It was in common use around the southern shores of the Caribbean Sea from Guiana to Yucatan with the greatest concentration in Panama and Colombia.

Sir Walter Raleigh, on his voyage to Guiana in 1595, when 150 miles up the Orinoco asked a native chief how they made their golden plates.[2] The chief replied, "They gathered it (from rivers) in graines of perfect gold...and that they put to it a part of copper, otherwise they could not work it, and that they used a great earthen potte with holes round about it, and when they mingled the earth and copper together, they fastened canes to the holes, and so with the breath of men they increased the fire till the metall ran, and then they cast it into moulds of stone and clay, and so make those plates and images."

The objects made from this alloy (called tumbaga by the Spaniards) were gilded by the process known as *mise en couleur*. Gomara in 1552 described the process as he observed it in northern Colombia.[3] "There is much gold in Santa Marta and copper which they gild with a certain crushed and macerated herb. They rub the copper (tumbaga) with this and put it in the fire. It takes on more color the more herb they apply to it and it is so fine that in the beginning it deceived many Spaniards."

It was only after a long time that archeologists found examples of this type of gilding. They found objects of yellow and red gold but none of the alloy. They

[1] WAGNER, H. R., "The Discovery of New Spain in 1518 by Juan de Grijalva," Cortes Society, **1942**, p. 94.

[2] RALEIGH, W., "The Discovery of . . . Guiana," Hakluyt Society, **1848**, p. 93.

[3] LOTHROP, S. K., "Cocle," part 1. Memoirs of the Peabody Museum. Harvard University, **1937**, Vol. VII, p. 84

did find gilded objects of a soft black material which looked like some sort of organic material, and it was assumed that the Indians had carved their figurines from some such substance and then gilded them. About thirty years ago when the first large scale analyses of archeological metal objects were made it was found that this soft black material was a mixture of cuprite (Cu_2O), malachite, and powdered gold.[4] Furthermore, many of the objects that were thought to be of pure gold contained considerable amounts of copper. The objects with the black cores were those with much copper. In the course of 400 years they had undergone almost complete corrosion.

Rivet supposed that the alloy was used because it can be made to resemble gold, and what the Colombians went by in mixing alloys was the color of the resulting product.

Lothrop and Root[5] investigated a large number of metal objects from Coclé in Panama. They found many examples of *mise en couleur* but they also found when polished sections of some of the objects were examined that they had been gilded by the application of gold leaf to a base of tumbaga. It is possible that some of the objects thought to have been gilded by *mise en couleur* are actually leaf-gilded. It is almost impossible to tell them apart by visual examination of the objects. They came to no definite conclusions as to why tumbaga was used as an alloy.

In the twelve years since 1937 I have analyzed many more objects from Panama, Costa Rica, Guatemala, and Yucatan. Including the earlier ones from Coclé I found 93 examples of tumbaga. With the 35 examples from Rivet's work there is a total of 128 analyzed objects of this alloy. This is enough to make it worth while to consider again the question of why it was used so extensively by the natives of Central America and Colombia.

The physical properties of gold, silver, and copper alloys have been thoroughly studied by metallurgists. Gold melts at 1063°C. and copper at 1084°C. The eutectic contains 18 per cent of gold and melts at 880°C. or about 200° lower than the pure metals. The addition of silver lowers the melting point a little more (Figure 1).

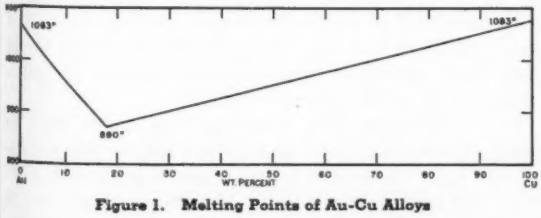


Figure 1. Melting Points of Au-Cu Alloys

Both gold and copper are fairly soft (about 50 on the Brinnell scale). With 25 per cent copper the hardness increases to 110. With more copper the hardness decreases (Figure 2). The addition of silver also increases the hardness to a marked degree. The hardest alloy of all contains 50 per cent gold, 25 per cent silver, 25 per cent copper. This has a hardness of 160, and on cold working this is increased to 200. This is harder than most Peruvian bronze (150).

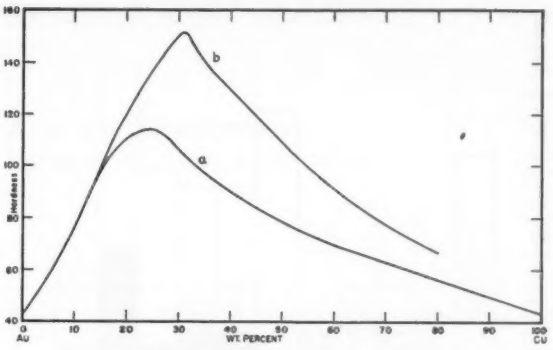


Figure 2. Brinell Hardness of Au-Cu Alloys

a, no silver; *b*, 20% silver

Gold containing even 10 per cent of copper is somewhat red. With 60 per cent or more of copper it is so red it looks almost like copper.

Bergsøe,[6] has carried out a series of experiments with various gold-copper alloys to see if he could gild them by the method described by Gomara. He heated the alloy to about 800°C. until it turned black through the oxidation of the copper on the surface. He then dipped it into an acid bath. The oxide dissolved leaving a gold surface. This could be repeated several times, the layer of gold becoming thicker with each treatment. With 50 per cent copper one heating was sufficient. With 75 per cent copper it required 3–4 heatings. With 90 per cent copper he obtained a gold layer but it flaked off. Thus this method of gilding is a feasible one for tumbaga with less than about 85 per cent of copper.

Figure 3 shows the number of objects of a given copper content for 5 per cent intervals for the 128 objects that have been analyzed. They are divided into four classes: (1) objects definitely gilded by *mise en couleur*, (2) objects gilded by the application of gold leaf, (3) objects that were left ungilded, (4) objects that were gilded but the method is uncertain.

The objects show great variation in composition. The peak falls between 30 and 55 per cent copper.

Most of the ungilded objects contain less than 40 per cent of copper. These constitute the "red gold" objects sometimes found. For the most part these have suffered little corrosion (Figure 4*a*).

Objects gilded by *mise en couleur* contain from 5 to 90 per cent of copper. The largest number fall between 30 and 65 per cent. Those rich in copper have undergone almost complete corrosion. It is interesting that three of the objects were more than 85 per cent

[4] Crequi-Montfort, G. de, P. Rivet, and H. Arsandaux, *Journal Soc. Americanistes de Paris*, **11**, 548–561 (1919).

[5] Ref. 3, pp. 78, 307.

[6] Bergsøe, P., "The Gilding Process and the Metallurgy of Copper and Lead among the Pre-Columbian Indians," Ingeniørvidenskabelige Skrifter Nr. A 46, Copenhagen, 1938, p. 37.

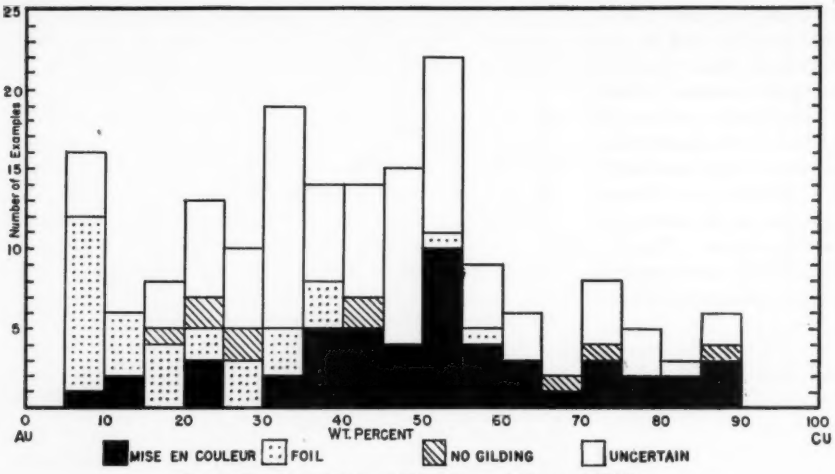


Figure 3. Composition of Tumbaga and Types of Gilding

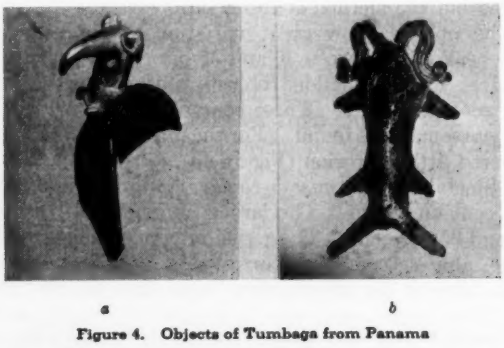
copper even though Bergsøe has shown that such an alloy cannot be successfully gilded by the process of *mise en couleur* (Figure 4*b*).

Objects gilded with gold leaf contain from 15 to 90 per cent of copper. The majority of them are rich in gold, several of them would fall in the region of red gold. Apparently it was almost as easy to gild by this method as by *mise en couleur*. The technique used is still obscure.

There are four reasons why tumbaga might have been used:

(1) It made casting easier.
(2) It made objects harder.
(3) It made a small amount of gold go further.
(4) The natives liked the color of gold better than that of copper.

Many objects of pure gold of very complex form have been found in Panama and Colombia in spite of the high melting point of gold. Alloys containing 30 per cent of copper melt about 140°C. lower; ones of 55 per cent copper melt 93°C. lower. Hence in spite of Raleigh's statement it does not seem that increased ease of casting was an important factor in the use of tumbaga.



a *b*

Figure 4. Objects of Tumbaga from Panama

a, Au 58%, Ag 2%, Cu 40%; "red gold." *b*, Au 17%, Ag 9%, Cu 74%; mise en couleur. Objects are in the Peabody Museum, Harvard.

Neither does increased hardness appear to have much to do with the use of tumbaga. Most of the objects of tumbaga are figurines, pendants, rings, bells, etc. There are very few chisels, knives, or axeheads. In Mexico many knives and axeheads have been found, but they are all of copper. Here was an opportunity to have a great increase in hardness through the use of tumbaga, but there is no indication that this was known.

The third reason, that the use of tumbaga permitted a little gold to go further, does not apply to Panama and Colombia where gold was abundant and copper was relatively scarce, but it might apply to Mexico where there was more copper than gold. Yet the great majority of objects of tumbaga are from Panama and Colombia. The few examples of tumbaga that have been analyzed from Mexico show much the same distribution of composition as is found in Colombia. One would expect that they would have a higher copper content than they do. Probably knowledge of tumbaga and *mise en couleur* came to Mexico from Colombia at a fairly late date, and their possibilities were never fully explored by the Mexican metal workers.

This leaves only the fourth reason as a likely one. The natives of Panama and Colombia, and of Mexico to a lesser degree, liked the color of gold better than they did that of copper. The use of tumbaga gave them a way to use the copper they had in the manufacture of metal objects which would still be gold in color. It appears doubtful if, in Colombia at least, there was any real attempt made to prepare an alloy of even roughly a given composition. It is probable that all of the available gold and copper was melted together, and then an object was cast in the usual fashion. If the final color was only a little reddish it might be left as it was. If it was too coppery it was then gilded by *mise en couleur*, or leaf-gilded if of a suitable shape. In this way all of the available metal was put to good use, and it would make little difference to the Indians whether the alloy contained 30 or 70 per cent of gold.

It is interesting that tumbaga was never used in Peru. On the northern coast, where copper was plentiful and gold was scarce, pure copper objects were covered with gold or silver leaf. Bronze was also used in later times. This would indicate little or no trade between Colombia and Peru.

The discovery of tumbaga was the greatest contribution of Colombia to the metallurgy of the New World. It was used primarily as a way of coloring copper, and while it could have been profitably used for other reasons there is little reason for thinking that it was.

CONSERVATION OF ANCIENT MARBLE MONUMENTS

A. J. SOFIANOPOULOS
University of Dayton, Dayton, Ohio

THE cultural interest associated with the conservation of the large stone monuments of past civilizations is very great. Unfortunately, many of these ancient structures weather at an accelerating rate, and it is to be feared that sooner or later the progress of deterioration may be beyond control and the state of the monuments beyond repair.

No systematic study of the mechanism of the weathering of ancient stone monuments has previously been made. Although something is known about the causes of the aging of rock in quarry strata, the aging process may often be somewhat different with stones exposed on structures. This is because new causes appear, especially when a modern city surrounds an ancient structure. Usually the problem of the conservation of ancient buildings has been entrusted to construction engineers, who have paid ample attention to the problems of mechanical repair but no attention to the underlying chemical causes of deterioration.

This paper deals with the weathering of marble from the chemical viewpoint, and also contains some remarks on the repair and restoration of ancient marble structures. Although the discussion is specifically about the monuments on the Acropolis at Athens, Greece, the conclusions appear to be applicable generally to similar structures standing under similar climatic conditions.

The marble used in the construction of these buildings at Athens is generally known as Pentelic marble, so named from its source, Mt. Pentele, about 12 miles northwest of the Acropolis. The ancient quarries were located at Spilia on the southern slopes of the mountain, and the workings, as well as a large portion of the ancient quarry road leading toward Athens, are still plainly visible today. This marble is nearly pure calcium carbonate, an average content of slightly over 99 per cent being indicated from analyses. Iron is present up to a maximum of 0.15 per cent in the form of ferrous carbonate and hydrated ferric oxide. Silica and silicate minerals are also present as impurities, sometimes in the form of visible inclusions. By simple inspection it is possible to select pieces which are essentially pure calcium carbonate. The specific gravity of this marble lies uniformly between 2.72 and 2.73, and its degree of water adsorption is 28 grams per square meter as determined by the immersion of slabs $18 \times 12 \times 2$ cm. in water for 120 hours under normal pressure. This means that on a building the stone is practically impervious to rain water and that mechanical damage due to freezing cannot occur in stones free from cracks. It is relatively easy to select large pieces of this marble that are very homogeneous both physically and chemically, and there is clear evidence that the ancient builders recognized the importance of such selection. On the Acropolis today are to be seen rejected stones that contain imperfections in the way of inclusions and cracks. It is significant that these stones are much more badly weathered than the stones in the buildings.

The marble in these buildings appears to have undergone no damage due to thermal fluctuations, and this lack of damage may be ascribed both to the homogeneity of the stone and the peculiar method of construction. In general, gross mechanical damage to the stone of the buildings throughout the centuries has been due to deliberate damage by man rather than natural causes.

Since calcium carbonate is appreciably soluble in water, especially water containing carbon dioxide, it might be supposed that the solvent action of rain water on the marble surfaces would by this time have resulted in severe damage by erosion. Actually, the damage from this cause has been relatively slight, and there is no evidence that the solvent action of rain water on *clean* marble surfaces is an important cause of deterioration.

The most conspicuous indication of the deterioration of the marble is the presence of discolored areas on the surface of the stone. The color of these is predominantly light brown, but in some places the color is a very dark brown and even black. It is in these areas that the marble has deteriorated the most. Here the surface of the stone is often rough; elsewhere, also, thin layers of stone split away from the surface, and even large pieces break away and fall. It has generally been supposed that the discoloration is the result of the oxidation of the ferrous iron in the marble to dark ferric oxides which deposit on the surface. But various facts are inconsistent with this as an adequate explanation. If mere oxidation were the full explanation one would expect the discoloration to be more or less uniformly distributed, whereas, as a matter of fact, it is very much localized, being in general confined to the upper and protected parts of the buildings. This localization of the discoloration cannot be ascribed to the use of marble blocks of different iron content, as some have suggested, since the iron content of this marble varies only within narrow limits as shown by the analyses. Furthermore, one side of a given block may be badly discolored and the other side may be of normal color. Even if some

sort of selective oxidation of the iron occurs it is not easy to understand how the ferrous iron in the rather impervious stone could become oxidized.

A careful examination of the discolored areas showed that they are coated with a thin layer of extremely fine dust containing clay, silica, soot, etc., as well as vegetable matter such as spores, and that the discoloration is only in part due to the presence of iron compounds. Evidently this material is in large part very fine atmospheric dust that has settled on these areas and there adhered. The thickness and position of these deposits of dust may be correlated with the direction of the local winds and rains, and the degree of protection from rain. No dust at all has adhered to marble surfaces directly subject to driving rain, and these are not discolored. In partly protected parts of the buildings the layer of dust is thin, but in well protected parts it has accumulated into relatively thick layers.

The presence of these layers of dust makes possible the following explanation of the deterioration of the marble in the areas where this dust has been deposited. This explanation is based both on the observed facts and accepted general chemical principles.[1] The fine dust is almost of colloidal dimensions and has a very large surface compared to its mass. By reason of this large surface an appreciable amount of atmospheric moisture can be adsorbed on the dust and firmly retained. This moisture contains in solution the various gases of the atmosphere, including oxides of nitrogen and particularly the sulfur dioxide present in the air of a modern city. These acid gases are oxidized by the dissolved oxygen, and nitric and sulfuric acids are formed in solution. In damp weather the amount of such adsorbed moisture may be relatively large, but with a succeeding period of dry weather it is reasonable to expect both a reduction in its amount and an increase in the concentration of the acids. Because of its composition this dust is not dissolved or otherwise affected by the acids, and it seems not at all unlikely that this may act as a contact catalyst in promoting the oxidation of the gases. Whatever may be the exact mechanism for the formation of the acids, they readily attack the marble, dissolving away the calcium carbonate and oxidizing the ferrous carbonate to ferric compounds. Some of these at least are insoluble or slightly soluble in the dilute acid solution and tend to accumulate on the surface of the stone. As the acids are neutralized in these reactions more are formed by the solution and oxidation of the gases, so that there occurs a continuous chain of chemical changes that causes the progressive deterioration and discoloration of the stone.

This kind of destructive action apparently proceeds at an even greater rate in the joints between blocks of stone and in cracks or crevices. Here considerable amounts of atmospheric dust may accumulate, greater amounts of acid are formed, and the rate of corrosion is consequently correspondingly greater. There is some indication also that the crystallization of soluble calcium salts, such as the sulfate, in crevices exerts mechanical destructive action similar to that due to the formation of ice. At any rate, pieces of stone have been observed to fall from the edges of joints or cracks during heavy rains as though some soluble cementing material was being leached out. The growth of vegetation is still another source of destructive action in joints and cracks. Here seeds take root in the accumulated dust and the growth of the roots causes fractures in the stone.

Various means have been suggested for treating marble to protect it from atmospheric action. For example, it has been suggested that the marble be treated chemically so as to form a superficial layer of a calcium compound more insoluble than the carbonate, as may be done by means of a soluble silicate solution. Though attractive in theory this type of treatment has not worked out well in practice, as the surface layer often has a tendency to separate from the body of the stone in the course of time, probably because of stresses set up by thermal fluctuations. More satisfactory is the application of linseed oil, and other drying oils, varnishes, waxes, or insoluble soaps as protective materials. The application of such materials to marble objects in museums has been widely practiced with very satisfactory results. For large stone structures, one important general objection to either of these types of treatment is the large expense involved, especially if treatment must be repeated at fairly frequent intervals, as seems almost certain to be necessary.

The only practical method for larger exterior structures appears to be to wash off the atmospheric dust, the principal cause of deterioration, before it can do any damage. In other words the best remedy is the very simple one of washing the marble periodically, as may be done conveniently in practice with a high velocity stream of water.

It is interesting to note that the ancient Greeks washed at least some of their temples periodically. This practice is definitely known for one of the temples on the Acropolis, namely, the Hecatompedon. According to the account that has come down to us, this temple was washed on a given day in the spring of each year by youths of Athens. The alleged reason for this washing was to remove a curse imposed on the temple because at one time its sanctity had been violated by the city magistrates of Athens when they ordered eviction and execution of conspirators who had sought asylum there. However, this periodic washing had the very practical advantage of keeping the temple clean, and also the advantage of promoting its conservation. It is highly probable that the first of these practical advantages was readily recognized and not improbable that the second may have become recognized in the course of the years. That the aspect of conservation was recognized to some extent is indicated by the treatment given at the same time to the wooden statue of Athena contained in the temple. This statue was carried to the sea at Phaleron and immersed in the water for an entire day. The practice of im-

[1] SOFIANOPOULOS, A. J., *Praktika of the Academy of Athens*, **13**, 775 (1938); **14**, 398 (1939); **15**, 196 (1940).

mersing wood in sea water to promote its preservation appears to have been widely practiced by the ancient Greeks, according to Theophrastus,[2] who terms this procedure the embalming of the wood. It may well have been that the practical considerations of cleansing and conservation were real bases for the long continued practice of washing the temple and its statue annually, even though this practice was supposed to have a religious basis only.

Chemical considerations also enter into the repair and restoration of ancient marble buildings. Here the principal problem is the selection of proper cements or mortars for fastening broken blocks together or for patching surfaces so as to restore them to their original smooth condition. It might be supposed, offhand, that this would be a simple problem in view of the variety of strong hydraulic cements, such as portland cement, and the variety of special mortars that are now available. However, experience[3] has shown that these are not at all satisfactory for this particular purpose. Some of the modern cementing materials that have been used for the repair of the temples on the Acropolis in recent years are in a far worse state of preservation than the marble to which they have been applied. Many appear to lack durability from the chemical standpoint when thus measured in terms of years or decades, and from the physical standpoint they are too different from the marble to which they are applied so that separation takes place sooner or later under the action of thermal fluctuations. In contrast to this, most of the ancient repairs made centuries ago are still in perfect condition, even though the layer of cementing material is often very thin. The proper cementing material appears to be one that resembles as closely as possible the stone itself, both chemically and physically. The scientific examination of satisfactory ancient mortars has shown this to be so. As an example may be cited the results of an examination of the mortar on some column drums lying scattered west of the Parthenon. These drums, which are of limestone, are attributed to a prehistoric temple, and the mortar was evidently applied to smooth out the irregularities of the limestone surface. The aggregate of this mortar consists of fine particles of broken limestone, not silica sand, and the chemical analysis showed that the mortar as a whole consists of calcium carbonate, together with small amounts of insoluble matter, iron oxide, and aluminum oxide. In other words its chemical composition was very similar to that of the limestone itself. This mortar would appear, therefore, to have been originally a simple lime mortar with limestone particles as the aggregate. However, this does not explain adequately the hardness and great durability of this mortar. One other fact is of importance, namely, that the mortar was found to carbonize on heating, thus indicating the presence of organic matter. Other ancient Greek mortars, for example specimens from the joints of ancient tile water conduits in Athens and Marathon, were observed to behave in a similar manner on heating. Though it might reasonably be concluded that such organic matter had infiltrated into the mortar after it had hardened, it is much more probable that this was present as an original ingredient. Indeed we have definite evidence that colloidal organic matter was incorporated into lime mortars in former times. Codinos, an eye witness to the construction of the famous church of St. Sophia at Constantinople states in regard to the preparation of the mortar, "They were boiling barley in large pots and used with the lime barley extract instead of water, not too warm or too cold, in order to produce a cement that was as hard as iron." It is a fact that that mortar is still very hard and in very good condition. This seems to be the clue to the good state of preservation of ancient Greek mortars in general. Modern experience also shows that the incorporation of organic material into lime mortar has a beneficial effect. Such materials as sugar and skim milk have been frequently used, these forming to some extent saccharates or lactates of calcium. However, colloidal organic matter appears to have an especially beneficial effect in regulating the rate of drying and setting of mortar. The theory of this may be briefly stated as follows. Adequate hardening of lime mortar depends upon the conversion of a large proportion of the calcium initially present as the hydroxide into calcium carbonate, and also on the formation of long intertwined crystals of the carbonate. The carbonate is only so formed as the result of a reaction between the carbon dioxide in the air and calcium hydroxide in solution in the water of the mortar. However, the water may evaporate from an ordinary lime mortar before much of the calcium hydroxide has been converted to the carbonate. The result is a weak, crumbly mortar having little durability. But with a colloid present to bind the water the rate of evaporation is much slowed down and then a large proportion of the hydroxide is converted to the carbonate, thus resulting in the formation of a hard durable mortar.

The proper mortar for the repair and restoration of ancient marble monuments would appear, therefore, to be one with an aggregate consisting of crushed marble of the right particle size, together with the correct proportions of water, slaked lime, and colloidal organic material. It is still uncertain as to what colloidal organic material will give the best results and what the optimum proportion is. Some systematic experiments are now needed in order to determine the exact formula for the best mortar to be used in the repair and restoration of these invaluable ancient structures.

ACKNOWLEDGMENT

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THE COMPOSITION AND WORKING PROPERTIES OF ANCIENT GLASSES

FREDERICK R. MATSON
The Pennsylvania State College, State College, Pennsylvania

GLASS objects of many kinds—beads, cups, vases, lamps, even funerary urns—have been found among the remains of ancient civilizations of the Old World. Far more fragments than whole pieces have been discovered; together they demonstrate the multiplicity of shapes, colors, and decorative processes that were employed by ancient glassmakers and give clues as to the techniques of manufacture used in different regions and at different times. About 150 chemical analyses have been made of glass fragments that could be spared from collections during the past hundred years; 235 spectrographic analyses and a few optical measurements and physical tests have now supplemented these data so that together they show the major trends in early glass compositions. The purpose of the present paper is briefly to summarize the literature available, to indicate the more common types of compositions used in Egypt, Rome, the Near East, and China together with the evidence they give of the raw materials used and the working properties of the glasses, and to suggest nondestructive tests that could rapidly increase the useful information available.

LITERATURE

The outstanding work on ancient glass compositions is that of Neumann. His assistants analyzed 69 glass specimens obtained through museums from archaeological sites in Egypt, Mesopotamia, and Roman Gaul, and he interpreted the results, including in his report 33 analyses published by other workers and a study of sources of soda available to the glassmakers.

The material remains of ancient Egypt have been more extensively analyzed and studied than those from any other region. The late A. Lucas, at one time Director of the Chemical Department of Egypt, worked for many years in this field. In three successive editions of his book, "Ancient Egyptian Materials and Industries," he brought together all the available technical information. The chapter devoted to glass and glass making includes chemical analyses of glasses published by Parodi, Neumann, and Lucas' own staff, as well as good discussion of raw materials and their sources.

A useful source book is Partington's "History of Applied Chemistry." Glass is discussed in each of the geographical sections of the volume, but the only useful analyses given are those by Neumann. Unfortunately most reports by earlier authors do not describe the glass and its provenience in sufficient detail so that it can be dated and used with later data. Therefore most of the analyses published prior to 1925 are of secondary value.

Two papers on the spectrographic analysis of ancient Chinese glass by Ritchie and by Seligman and Beck and one on Egyptian glass by Farnsworth and Ritchie contain nondestructive analyses of 235 pieces of glass. Studies on such a large scale made possible the comparison of some types of Oriental and Western glass, the tracing of major changes in glass composition in China, and the determination of the colorant in the blue glasses of Egypt and the East. The use of cobalt instead of copper to produce blue glasses has been a controversial point for a long time, and one that was not satisfactorily solved by the more usual analytical procedures.

Several papers have dealt with specific glass problems or have included a chemical analysis of glass in the general text. These will be mentioned in the regional discussions of glass compositions.

COMPOSITION

The general field of ancient glass compositions other than those of China can be represented by the range in oxide values shown in Table 1, which does not necessarily include the extremes. For comparison the general range in modern commercial plate, window, container, and bottle glass (omitting lead glass) is shown in a parallel column.

TABLE 1
Range in Compositions of Ancient and Modern Glasses

	Ancient, %	*Modern, %*
SiO_2	57–72	63–74
Al_2O_3	0.6– 5	0– 5
Fe_2O_3	0.2– 3	0.2– 2
CaO	3–10	3–15
MgO	0.2– 5	0.3– 5
Na_2O	9–21	6–22
K_2O	0.2–3	0.4–3

A glass consisting of 100 per cent SiO_2 would be ideal in many respects, but it melts at much too high a temperature (1713°C.) to be practical for most purposes. The addition of Na_2O rapidly lowers the liquidus temperature, the eutectic mixture of the two having a melting point of 793°C. Unfortunately sodium silicates are soluble in water, so the glasses need a stabilizer, and CaO is added for that purpose. The eutectic mixture of this three-component system has a melting point of 725°C., but higher working temperatures are needed to reduce sufficiently the viscosity of the glass.

The range of possible glass compositions in the soda-lime-silica system is quite limited, as Morey has shown by means of the ternary phase diagram. If more than about 74 per cent SiO_2 is used the glass is too viscous to work at normal temperatures, if too much CaO is used the glass is not only hard to melt but will tend to devitrify, and if there is an excess of Na_2O present the glass will be readily attacked by water. The ancient glass that exists today does not necessarily represent the entire composition range used in the past, for only the more durable glasses with moderate alkali content have survived the moisture attack during burial.

The analyses shown in Table 2 have been selected to show some of the more typical compositions used, in so far as they can be so termed with the data now available. The major components are grouped together for each glass in the upper lines of the table so that compositions can be easily compared. Below these summaries appear the detailed analyses.[1]

The eutectic mixture in the soda-lime-silica system is approximately: SiO_2, 73 per cent; CaO, 5 per cent; Na_2O, 22 per cent. It will be of interest to keep this in mind when examining the analyses reported in Table 2.[2]

Egyptian glass is documented with 64 analyses in the literature. From these, 6 have been included in Table 2 to show the variation that occurred. One Roman glass to represent the approximately 40 published analyses and a late Babylonian glass are also listed.

WORKING PROPERTIES

It is apparent from the great difference in the proportions of silica, alkaline earths, and alkalies in these eight glasses that their working properties would be quite different. This may not be as important a point as it would at first seem, because the viscosity of the glass could be reduced by raising the temperature in the furnace. Thus the less fluid glasses could still be worked if the furnace were suitable and enough fuel available. Besides viscosity, the rate at which a glass will set up as it cools is an important working property. To a large extent it is controlled by the alkaline earth content. It may be quite fluid at the higher temperatures, but will stiffen very rapidly as it cools if it is high in lime, whereas glasses that are equally fluid at high temperatures but are lower in lime will not set as rapidly. For this reason the modern glass compositions used for making plate and window glass are higher in lime than those for glass which is to be blown into the desired shape. The earliest glass vessels were formed around cores of clay and sand and were decorated by the surface application of colored glass threads. Formulations that set up fairly quickly would be advantageous. When the technique of glass blowing was introduced around the first century B.C., however, it is probable that the compositions were changed to permit a longer working time of the glass without surface devitrification. The new compositions were higher in alkali and silica and lower in alkaline earths. Unfortunately, the data given with most of the published glass analyses are insufficient to trace the changes in composition as related to the technique of manufacture. However, it is apparent that most of the analyses of Roman glass from any part of the Roman Empire are quite similar to that of the Cologne specimen in Table 2. The wide variation in the composition of the earlier Egyptian glasses is probably the result of manufacture at different industrial centers in the Kingdom, each of which had its own craft traditions and special raw materials.

A guess can be made as to the general working properties of the glasses shown in Table 2 from a study of the essential part of the compositions. The colorants must also be kept in mind, however, for the depth of color will affect the temperature gradient in the glass, and each colorant of course participates in the chemical reactions that determine the working properties.

Glass No. 1 will probably be quite fluid at the higher temperatures but will set up rapidly as it begins to cool, due to the high $CaO \cdot MgO$ and R_2O_3. If it is held too long at a temperature a little above its softening temperature it will tend to devitrify. It would be difficult to eliminate the fine bubbles in the glass.

No. 2 would be easier to work, for its higher alkali content would make it less viscous, yet the alkaline earth content is still high enough to cause it to set up rapidly.

No. 3 with its extremely high alkali content should be a very soft glass, easy to work but quite susceptible to weathering. This is an old analysis (1908), and only one of Neumann's showed more than 22 per cent alkali, while 11 out of the 13 reported by Parodi had 28–30 per cent alkali, so his work may be open to question unless future studies produce similar results. It seems doubtful that such a highly alkaline glass would not decompose in 3000 years unless it were completely protected from moisture.

No. 4, a glass of the Hellenistic Period when the technique of glass blowing was introduced, differs markedly from 1 and 2 in its much lower alkaline earth (predominantly CaO) and higher silica. It is a composition that could have been used for blown glass and quite closely resembles the Roman glass from Cologne, No. 8.

No. 5 resembles 4 and 8 and would be a very good

[1] This form of presentation was chosen rather than a listing of the range from minimum to maximum for each of the oxides that compose the glasses because it should give a clearer picture. Range statements are misleading for they cannot show the simultaneous change in several variables. Neumann has plotted the compositions on triaxial graph paper, using SiO_2 plus R_2O_3, the alkalies, and the alkaline earths as his three variables. This is helpful in preliminary studies, but the grouping of SiO_2 and R_2O_3 together as one component can result in very misleading charts.

[2] Of course the addition of other ingredients will affect the working temperature of the glass, so the above tabulation can serve only as a rough guide. Neumann determined the "melting" points of 22 of the glasses that he had had analyzed and found that they ranged, in the test that he used, from 670° to 880°C. Further work should be done to determine the working properties of some of the glasses. Viscosity measurements might produce the most useful results, providing the temperature gradients in colorless and deeply colored glasses are kept in mind.

composition except for its high R_2O_3, which would tend to increase its viscosity. This glass should have very good chemical durability. This glass is reported to have come from the XII Dynasty Theban tombs, but if so, it may have been a late inclusion therein, for it greatly resembles many analyses of glasses made in the Hellenistic and Roman Period. Parodi reports two XII Dynasty analyses similar to No. 5 except for their higher alkali content. Were a detailed description of the shape and decoration of the fragments available, or were the nature of their discovery known, one could better judge whether a XII Dynasty date is acceptable.

No. 6 is unique in its low lime content. The small amount present might have come in as an impurity in the sand. The R_2O_3 is exceptionally high—it must have been a very stiff glass to work. This glass was used in the manufacture of glass weights which were stamped with an inscription, so a very viscous glass would be quite suitable.

No. 7 with its high CaO·MgO and low alkali must have been very difficult to work. There is a possibility that the weathered surface, low in alkali, of the glass fragment had not been completely removed, or that the sherd was very thin so that some of the alkali had leached out of the apparantly unattacked zone. There are too few analyses of Babylonian glass available to judge whether this should be considered a typical composition.

No. 8 represents the type reported with no great variation throughout the Roman provinces. It seems to be typical of blown glass for its proportions are such, aside from its high R_2O_3, that it should be a good workable composition for the glass blower.

RAW MATERIALS

The chemical analysis of a glass can supply some clues to the raw materials used, particularly if one is familiar with the composition of the local materials in the region.

Sand was used to introduce the SiO_2 into many of the glass batches. Contaminants such as feldspar and clay could account for much of the Al_2O_3 present and for some of the Fe_2O_3. Lucas reports that the sands of the northern shore of Egypt contain much $CaCO_3$; therefore, a low lime glass such as No. 6 in Table 2 may have had no intentional addition of lime. Crushed sandstone or quartzite, perhaps thermally fractured, may also have been sources of SiO_2.

The relative proportions of CaO and MgO will indicate the type of limestone used in the glass batch. A dolomite would have almost equal portions of both, while a fairly pure calcite would be very low in MgO. The relative proportions of the two characterize some limestones and may furnish a useful analytical device when the chemical nature of the local limestone is known. The CaO:MgO ratio is included in Table 2. It shows that four of the Egyptian glasses, Nos. 1, 2, 3, and 5, had been made with dolomitic limestone as a batch ingredient, while the other two, which came from the region around Cairo, were much higher, relatively, in CaO. This is in agreement with the work of Lucas, who found that the MgO content of most of the limestones around Cairo was low. Many of the analyses of Roman glasses from Gaul show that they are almost lacking in MgO. Since MgO causes a glass to set up much more rapidly than does CaO as the molten glass cools, it is of great interest that the blown glass of the Roman period has a high CaO:MgO ratio. This may indicate intentional selection from among the available limestones, even to the extent of commercial shipments of desired types.

The Na_2O:K_2O ratio is also a useful value to consider. If Egyptian natron, a natural mixture of Na_2CO_3, $NaHCO_3$, NaCl, and Na_2SO_4, was used in the batch, then the K_2O content of the glass would be extremely small. Three such examples, Nos. 4, 6, and 8, are included in Table 2. Natron was an important commercial item in antiquity as well as today and was exported. When plant ash was used as the source of alkali, a small amount of K_2O is found in the glass, the amount varying with the nature of the plants. It is possible that some natron was also added to such batches, or that natron was used in one and plant ash in the other of two intermediate glasses that were remelted in the preparation of the final one. This study of analyses for clues to the raw materials used in ancient glassmaking is discussed in more detail elsewhere. A very high K_2O content at the expense of Na_2O is most unlikely. An analysis of a Roman glass, made by Manley prior to 1912 and republished by Caley because it contained uranium oxide, shows a K_2O content of 20.38 per cent and a summation for the analysis of 100.00 per cent. It is likely that Manley obtained his alkali by difference and incorrectly assumed that it was potash. Most Roman glasses contained very little K_2O with a maximum amount of 2 per cent, the greater portion of the alkali being Na_2O.

The amount of R_2O_3 in a glass will serve as one clue to its chemical durability; the greater the amount present, up to about 4 per cent with the alkali content remaining about constant, the better will be its resistance to chemical attack by water. By modern standards the glasses discussed are far too high in Al_2O_3. Probably partial solution of the walls of the clay crucible in which the glass was melted and the presence of very fine particles of unmelted batch in the opaque glasses account for this high Al_2O_3.

The colorants or opacifiers present in the glass and their relative amounts may provide useful clues if it is remembered that the glasses studied are mixtures of two or more intermediate glasses and that SnO_2 as an opacifier and the CuO, Fe_2O_3, or MnO as colorants may have been supplied by different intermediate glasses.

Even with a knowledge of the raw materials used, it is seldom possible to calculate a batch composition with any degree of accuracy except, perhaps, when a glass factory is studied such as that found by Petrie at Tell el Amarna. When a batch was melted in a clay crucible and then cooled and broken open, it is probable that only zones within the batch had become glass, and these were full of bubbles and streaked in color. After

the glass was separated from the surface scum and the unmelted batch, it could be used as one ingredient in a final glass composition. Thompson's translation of the Babylonian glass formulas recorded on cuneiform tablets indicates that at least two special glasses of different compositions and colors were combined with additional ingredients to form the final glass.

REGIONAL COMPOSITIONS

Egyptian glass has already been discussed in sufficient detail for this paper. Chemical analyses have been published by Parodi, Lucas, Neumann, Jackson, Collie, and Matson. Spectrographic analyses appear in papers by Seligman and Beck and by Farnsworth and Ritchie. Beck has also made a microscopic examination of ancient glass and has determined its specific gravity. In Mond and Myer's monograph Bannister determined the index of refraction and specific gravity of a series of early beads and plotted the values on a chart of the type prepared by Bannister.

Analyses of Roman glass are widely scattered in the literature. Neumann has published the most comprehensive series and has included some by earlier authors. One or more analyses also occur in the following papers and are frequently termed just "Roman glass" with no provenience or date: Pellatt, Sauzay, Turner, Laubengayer, Foster, Caley, and Matson.

Little has been published on the chemical composition of early Near Eastern glasses, although at present it would appear that the art of glass making originated in that region. Far less glass is known from Mesopotamia than from Egypt, in part because of the corrosive action of moisture and salts in the Near Eastern soils upon glass and also because much less controlled excavating has taken place there than in Egypt. A small clear glass cylinder was found by Frankfort at Tell Asmar, near Baghdad, that dates from the Akkadian Period, 2700–2500 B.C. It has an index of refraction of 1.51 and a specific gravity of 2.45, according to Beck, so was probably a simple soda-lime-silica glass. Work on an early glass found at Tell Judaidah, near Antioch, will soon be published by Dr. and Mrs. R. J. Braidwood. A glass bead from Megiddo in Palestine from the Middle Bronze II Period (about 1800–1600 B.C.) has been studied by Morgan and reported by Guy and Engberg. Rathgen analyzed a Babylonian vase. Neumann has published eight compositions of glass from Nippur, of the first few centuries B.C., and five from Samarra of the ninth century A.D. The three spectrographic studies already mentioned contain some examples from this region. There is much yet to be learned about Near Eastern glass, particularly in relationship to the compositions of the Mediterranean area and to the glazes that were developed.

Chinese glasses have been analyzed chiefly by spectrographic means because very little material can be sacrificed for examination and because of the many obvious advantages of this method of surveying a large quantity of material. It is hoped that chemical analyses will be made later of pieces that are established as truly representative of the period studied not only in shape, color, and design, but also in optical, physical, and spectrographic properties. Ritchie has shown that in pre-Han and Han times (prior to 200 A.D.) PbO-BaO-SiO_2 glass compositions were used. These gradually changed to PbO-Na_2O-SiO_2 glasses and finally in the T'ang Dynasty (600–900 A.D.) the Na_2O-CaO-SiO_2 compositions that had been long known in the West were in use. The lead-barium silicate and the lead silicate glasses could be easily melted and were used to form beads and small decorative objects. It is probable that these glasses were first formed as slags in the smelting of metals, so the two crafts were intimately related. Trade relations with the West gradually influenced the glass compositions used. A bead of Han times that contained 65 per cent PbO is reported by Seligman and Beck. While such a glass would be brilliant in appearance, it could be scratched or abraded easily and would develop a surface film due to moisture attack in a few years if it were not carefully preserved.

In Neumann's series of analyses lead was only occasionally found, and then in small amounts with a maximum of 1.3 per cent except in a special "sealing-wax-red" glass. Collie reports 31 per cent PbO in one such glass, which is colored by copper. The development of lead glazes is undoubtedly related to glass production and possibly to Chinese influence. That, too, is a problem which needs further study as well-excavated materials become available.

ANALYTICAL TECHNIQUES

Besides chemical and spectrographic analyses, spot tests for the presence of specific elements such as lead, copper, manganese, or cobalt can be used, and micro or semimicro analyses can be made. An examination of glass under a binocular microscope may indicate the technique of manufacture and will show the presence of unmelted batch materials, the size distribution of bubbles and striae or inhomogeneous streaks in the glass, and the degree of surface weathering that has occurred. The index of refraction can be measured on a very small fragment of glass, providing color streaks do not occur in the sample to cause index variations. This index is very sensitive to composition change; the same is true of the specific gravity of glass. The two values together often make possible the comparison of a series of specimens before a piece is selected for analysis. This was done by Matson (1948). The preparation of synthetic glass melts based on the analyzed compositions will aid in an understanding of the working properties, and viscosity measurements at a few temperatures would make a quantitative statement of setting rate of the glass possible.

Some analyses of ancient glasses are useless because the weathered surface layers (hydrated silica) or surface contaminants such as clay or metal had not been adequately removed before the analysis was made. In some, MgO and K_2O were not sought for. In some cases the specimens analyzed were poorly identified pieces of no direct value to museums.

Further chemical analyses of ancient glasses can be very useful if they are made on samples established as representative, if all the possible oxides present are sought for, if the sample is of definite provenience and can be dated, and if it is described in detail as to color, shape, and method of manufacture before it is crushed for analysis. When possible, one fragment from a series belonging to the same object should be chosen for analysis, for then pieces would still be available for reference after the chemical study had been completed.

Ideally, the glass from a site should be sorted within chronological periods as to method of manufacture, shape, and color, then checked by means of index of refraction and specific gravity measurements, and then analyzed statistically. Finally, on the basis of these data, samples should be selected for chemical analysis so that the results will truly represent the compositions used. This has seldom, if ever, been done, so the compositions available at present can only serve as an approximate guide for future work. Chemists and ceramists who are interested in archaeology as a hobby can use their laboratory skills and equipment on materials available in the storage rooms of museums and can thus greatly aid in tracing the technological development of glassmaking.

TABLE 2
Typical Analyses of Egyptian, Roman, and Babylonian Glass

	1	*2*	*3*	*4*	*5*	*6*	*7*	*8*
Essential Composition								
SiO_2	61.7	63.9	60.1	66.3	67.8	71.4	64.4	68.9
R_2O_3	3.1	1.3	3.1	4.1	5.5	6.7	4.0	4.6
CaO·MgO	15.1	12.1	5.7	8.6	6.3	3.5	12.1	7.9
Na_2O·K_2O	19.2	23.5	30.5	19.7	16.0	17.3	14.8	19.0
Ratios								
CaO:MgO	2	1.9	1.9	4.7	1.7	3.4	1.6	5.6
Na_2O:K_2O	11	28	21	48	6	57	6	94
Complete Analyses								
SiO_2	61.7	63.9	60.1	66.3	67.8	71.4	64.4	68.9
Al_2O_3	2.4	0.6	3.1	3.3	4.4	4.7	2.6	3.9
Fe_2O_3	0.7	0.7		0.8	1.1	2.0	1.4	0.7
CaO	10.0	7.9	3.7	7.1	4.0	2.7	7.5	6.7
MgO	5.1	4.2	2.0	1.5	2.3	0.8	4.6	1.2
Na_2O	17.6	22.7	29.1	19.3	13.7	17.0	12.7	18.8
K_2O	1.6	0.8	1.4	0.4	2.3	0.3	2.1	0.2
MnO	0.5	Trace	0.4	0.6	1.1	0.3	2.7	Trace
CuO	0.3	...	...	0.9	2.0	0.1	1.2	...
SO_3	...	...	...	...	1.0	0.1	1.0	...

1. Egyptian, XVIII Dynasty (1570–1346 B.C.), dark blue, opaque. From Tell el Amarna. Neumann #1.
2. Egyptian, XVIII Dynasty, colorless, transparent. From Tell el Amarna. Neumann #10.
3. Egyptian, XX Dynasty (*ca.* 1250–1150 B.C.), blue. Parodi, republished by Lucas.
4. Egyptian, 200–100 B.C., dark blue, transparent. From the Island of Elephantine. Neumann #21.
5. Egyptian, XII Dynasty (1989–1776 B.C.), dark blue, opaque. From the tombs at Thebes. Neumann #72. The date would seem to be much too early, judging from the composition.
6. Egyptian, Arabic, eighth century A.D., dark blue-green. From Cairo. Matson #34.
7. Babylonian, 250 B.C., dark blue. From Nippur. Neumann #93.
8. Roman, first century A.D., bluish. From Cologne. Neumann #34.

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ANCIENT SYRIAN COPPERS AND BRONZES

ROBERT J. BRAIDWOOD, JOSEPH E. BURKE, and NORMAN H. NACHTRIEB
University of Chicago, Chicago, Illinois

COPPER or "bronze" (although not in the strict sense in which we now use the term) is traditionally considered to be the oldest metal known to man. The Greek Epics speak of times when "man used weapons of bronze and wrought with bronze; for black iron was not." Lucretius (*ca.* 98–55 B.C.) says: "The earliest weapons were the hands, nails, and teeth; then came stones and clubs. These were followed by iron and bronze, but bronze came first...." The earliest "modern" archeological classification, published by C. J. Thomsen of the Royal Danish Museum in 1836, was essentially that of Lucretius: Thomsen called for "Ages of Stone, Bronze, and Iron" in that chronological order. In most parts of Eurasia, subsequent excavation has substantiated Thomsen's classification.

It is not yet certain exactly where and when copper tools were first made. There is enough certainty, however, for the assertion that the first use of copper was undoubtedly somewhere within the hilly flanks of the "fertile crescent" in the Near East (Figure 1). The date of its first appearance cannot be much later than *ca.* 5000 B.C. In our present state of knowledge, the earliest copper tools would appear to be those from the basal layers of the mound of Sialk in Iran.[1] By the time another thousand years had passed, simple copper tools and ornaments had made their appearance in Egypt (at Badari, near Tasa, see Figure 1), in Northwest

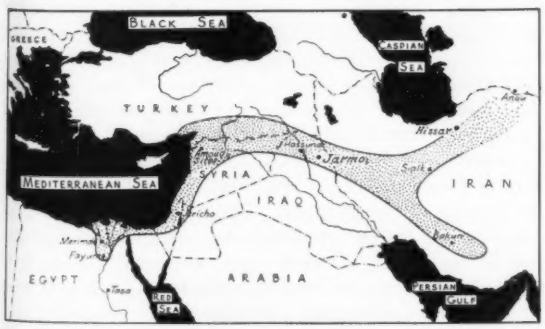

Figure 1. The Near East

Shows certain important archaeological sites in the earliest village stage (after 5000 B.C.) and emphasizes the hilly flanks zone which lay just above Breasted's "fertile crescent."

[1] The Sialk examples consist of pins and twists (earrings ?) of copper "wire," and of drills or reamers. They have not yet been given a full-scale technological report, but see H. H. COGHLAN, "Some fresh aspects of the prehistoric metallurgy of copper," *Antiquaries Journal*, **22** (1942). V. Gordon Childe's Huxley lecture, "Archaeological ages as technological stages," *Journal Royal Anthropological Institute*, **74** (1944) is a provocative account of the general archeological problems connected with early metallurgy.

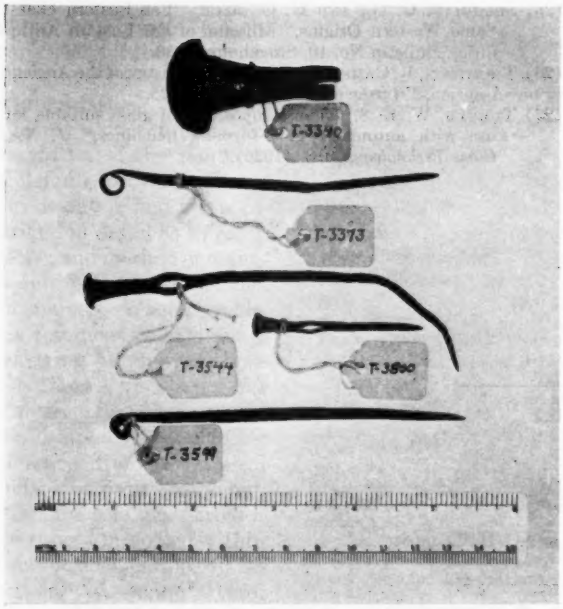

Figure 2. Collection of Pins Examined Chemically and Metallographically

Syria, and at other Iranian sites (Bakun, Hissar, and Anau).

The mineral sources of early copper are largely unknown, although it is certain that the mines of Cyprus were worked for native metal at a relatively early date. The present-day copper resources of the world give no clue to the ancient mines, which could scarcely be of current economic interest.

Other questions concerning the early use of copper have not been definitely answered. If, as Coghlan believes (and as in the New World, with Keewanaw ore), the first implements were fashioned of native copper,[2] then it is reasonable to ask how and where the knowledge of smelting arose. It would be interesting to know whether the reduction of ores and the fusibility of copper were simultaneous discoveries, or whether it was common practice to melt and cast native copper. The extent to which hot forging of metal was practiced would shed some light on the degree of knowledge possessed by the early metallurgists. A significant contribution would be the certain distinction between intentional alloys and natural alloys.

Qualified answers to some of these and related problems may be sought in chemical and metallographic examinations of the artifacts. Data of this kind are very meager and come from scattered sources, however. The sampling difficulties are large and the degree of accidental variation of metal composition is hard to assess. Enduring conclusions and valid correlations must be based upon the statistical examination of a large number of objects in the geographical and chronological ranges of interest.

The Oriental Institute of the University of Chicago has made available about 140 copper and bronze artifacts recovered from two sites in the Amouq (the plain of Antioch) in Northwest Syria by the 1932–38 Syrian Expedition. In the Amouq sites (see Figure 1) the cultural sequence from *ca.* 5000 to 2000 B.C. appeared as a succession of ten phases, designated by the expedition as phases A through J. Copper tools made their first appearance in phase F (*ca.* 3500–3100 B.C.), although some pieces came from a disturbed context, called the "First Mixed Range," which lay between phases C and F in one of the operations. By phase G (*ca.* 3100–2800 B.C.), contemporary with the first Egyptian dynasty, a fairly elaborate industry in metal is attested.

The First Mixed Range supplied a number of pin-like copper artifacts which we have called "reamers" for want of a better name, and reamers as well as more elaborate artifacts, such as needles, chisels, pins, maces, knives, and small human figurines were found in the later phases.

Chemical analyses have been performed on about twenty of the 140 specimens. Six reamers selected from phases F through I, as well as the First Mixed Range, were subjected to detailed metallographic examination since they represent a single industry whose manufacture was continued through a long period of time and because a great deal of information can be obtained about the method of fabrication by examining the microscopic structure of metals which have been mechanically worked. In addition, six bronze figures from phase G were studied to decide upon the method of casting.

CHEMICAL ANALYSIS

Qualitative spectrochemical examinations were made of all of the objects for the purpose of obtaining a general survey of chemical composition. Order of magnitude estimates were made of the trace and minor element concentrations. These proved to be useful in formulating methods of subsequent quantitative wet chemical determinations. In the series of twenty-seven reamers examined (First Mixed Range and phases F, G, H, and I) arsenic and nickel were found to be minor constituents (0.1 to 2 per cent) in all but one. Tin was absent in eighteen of the reamers. The nine reamers from the First Mixed Range and phase F were spectroscopically free of tin. This element varies erratically from 0 to 8.5 per cent in those corresponding to phases G, H, and I. It thus appears that a tin-free source of copper was available prior to phase G and that the alloying of this metal with tin occurred in the later periods. No correlation is apparent between the age of the reamers and their content of either silver or lead, which varies erratically from 0.00 to 1 per cent in the copper. Lo

[2] So far as we know, such a practice has not yet been definitely established for implements found in the ancient Near East. A good beginning on the problems involved here is indicated by two recent papers in *Man*, 48 (1948), nos. 3 and 17, entitled "Ancient mining and metallurgy group: preliminary report," Parts I and II, by Coghlan, Childe, Bromehead, and Voce.

concentrations of gold, bismuth, cobalt, iron, antimony, vanadium, and chromium appear in some of the reamers. There is some possibility that these elements may be used to trace the origin of the metal when data become available for the composition of Near East copper ores. Such metals as these are rather common trace constituents in many ores, however, so that their usefulness in this connection may be illusory. It seems more likely that the presence or absence of *significant* concentrations (0.1 to 10 per cent) of such elements as arsenic, nickel, and tin may be a more reliable clue to the origin of the copper.

Quantitative wet methods of chemical analysis were carried out on some of the reamers, other implements, and figurines. Conventional methods of analysis were employed and need merely be named. Copper was determined electrolytically; lead was determined either by weighing as lead sulfate or by the electrolytic method. Large amounts of arsenic were determined by weighing of arsenic pentasulfide, while trace amounts were

Figure 3. Collection of Reamers examined chemically and metallographically

determined by the molybdenum blue method after distillation as arsenic trichloride. Nickel was determined by precipitation with dimethylglyoxime. Tin was weighed as stannic oxide after ignition of metastannic acid. In a few cases silicon was determined from the loss in weight of ignited mixed oxides on treatment with hydrofluoric acid. Carbonate was determined on a few highly corroded specimens by evolution of carbon dioxide, as further described below.

The state of preservation of the artifacts, together

TABLE 1
Analysis of Fragments of Clay Crucible (X-3024), Phase G

Component	*Percentage*
SiO_2	50.55
Na_2O	2.60
R_2O_3	24.91
$CaO + MgO$	15.68
CO_2	4.06
Ign. loss $(-CO_2)$	2.21
	100.01

TABLE 2
Analysis of Charcoal Fragments (X-3100), Phase G

Component	*Percentage*
Cu (as CuO)	14.4
Fe_2O_3	0.5
SiO_2	11.3
H_2SO_4 – insol.	0.3
Al_2O_3	21.6
Ign. loss	56.8
	104.9

As and Ni detected spectroscopically (estimated 0.04%)

TABLE 3
Chemical Analyses and Hardness of Reamers

Element	*1*	*2*	*3*	*4*	*5*	*6*
Cu	95.53[a]	94.12[a]	93.73[a]	96.10[a]	93.99[a]	88.51[a]
As	1.35[a]	2.05[a]	0.45[a]	0.40[a]	1.62[a]	0.04
Ni	0.93[a]	2.73[a]	1.20[a]	0.39[a]	2.73[a]	0.16
Ag	0.005	0.005	0.005	0.005	0.005	0.005
Bi	0.001	N.D.	N.D	0.001	0.01	0.001
Pb	0.003	0.001	0.003	0.03	0.01	0.01
Si	0.001	0.001	0.01	0.05	0.01	0.05
Sn	N.D.	N.D.	1.52[a]	N.D.	N.D.	8.51[a]
Co	0.05	N.D.	N.D.	N.D.	0.02	N.D.
Zn	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
R_F[b]	102	50	87	99	93	108

[a] Wet analysis.
[b] Hardness, Rockwell F. Scale.
N.D.: Not detected in spectrographic analysis.

TABLE 4
Analysis of Fragment of Knob-Head Pin (X-2742a), Amouq Phase G

Constituent	*Core*	*Crust*	*Composite*
% of total	36.25	63.75	100.00
Cu	94.10	...	34.12
Ni	0.12[a]	0.003[a]	0.04[a]
As	0.53[a]	0.10[a]	0.26[a]
Fe	0.78[a]	...	0.28[a]
Insol. oxides	0.51	...	0.19
Moisture, 110°C.	...	0.09	0.06
Ign. loss, 700°C.	...	16.17	10.28
Malachite	...	2.08	1.32
Cuprite	...	82.94	52.85
	96.05	101.39	99.40

[a] Colorimetric.

TABLE 5
Analysis of Long Pin (X-2962), Amouq Phase G

Constituent	*Core*	*Crust*	*Insoluble*	*Composite*
% of total	13.47	80.66	5.78	99.91
Cu	96.44	...	...	13.00
Fe	2.15	...	...	0.29
Pb	0.74	...	...	0.10
As	0.59[a]	...	...	0.08[a]
Ag	0.07[a]	...	...	0.01[a]
Au	...	...	0.35[a]	0.02[a]
Sb	...	...	...	0.01
Mg, Mo, As, Ca, Ni, Bi, B, Co, Cr	...	...	1.02[a]	0.04[a]
SiO_2	...	1.09	86.50	5.88
SnO_2	...	...	7.79	0.45
Al_2O_3	...	1.45	2.60[a]	1.32
Fe_2O_3	...	1.10	0.87[a]	0.94[a]
TiO_2	...	...	0.87[a]	0.05[a]
Malachite	...	41.28	...	33.30
Azurite	...	Trace	...	...
Cuprite	...	36.02	...	29.05
Moisture, 110°C.	...	1.66	...	1.34
Ign. loss, 700°C.	...	17.39	...	14.03
	99.99	99.99	100.00	99.91

[a] Spectrochemical analysis.

TABLE 6
Analysis of Long Pin (X-3054), Amouq Phase G

Constituent	*Core*	*Crust*	*Composite*
% of total	30.88	69.12	100.00
Cu	98.96	...	30.56
Fe	...	...	...
Pb	0.30	0.11	0.17
As	0.49	0.48	0.48
Ag	...	...	...
Au	...	...	...
Sb	...	...	...
Ni	0.32	0.18	0.22
Insol. oxides	...	1.41	0.97
Malachite	...	9.98	6.88
Cuprite	...	80.65	55.75
Moisture, 110°C.	...	0.50	0.35
Ign. loss, 700°C.	...	5.00	3.45
	100.07	98.31	98.83

TABLE 7
Analysis of Long Pin (X-3311), Amouq Phase G

Constituent	*Core*	*Crust*	*Composite*
% of total	98.52	1.48	100.00
Cu	97.71	...	96.23
Ni	0.37[a]	Trace	0.37
As	0.50[a]	0.003[a]	0.49
Fe	0.01[a]	...	0.01
Insol. oxides	0.43	2.96	0.46
Cuprite	...	95.08	1.41
	99.02	98.04	98.97

[a] Colorimetric

with the qualitative spectroscopic information, determined the order and kind of determinations used. The condition of the specimens varied from almost completely coherent metal to a state of complete weathering—primarily to malachite and cuprite. In those cases in which there were appreciable quantities of both original metal (core) and enveloping corrosion products (crust) each fraction was separately analyzed. The entire sample was placed in an apparatus[3] for carbon dioxide determination and attacked with 6 *N* hydrochloric acid after air had been swept from the apparatus by means of a stream of purified nitrogen.[4] Malachite and cuprite dissolved, leaving the core of metal and a residue comprised largely of mixed oxides (SiO_2, SnO_2, Al_2O_3, and TiO_2). The percentage of malachite was

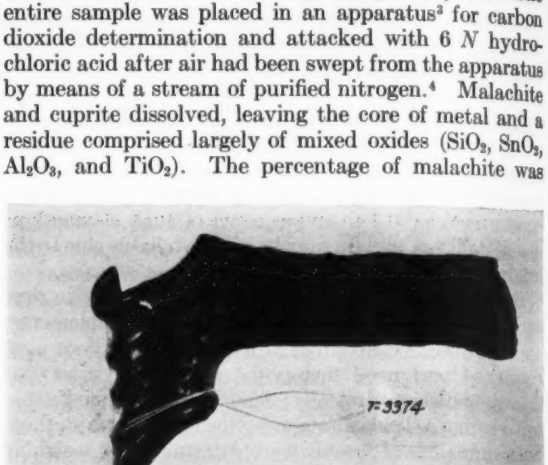

Figure 4. Axe head from Amouq Phase J

computed from the weight of carbon dioxide absorbed by ascarite in a gas-weighing bottle. The solution, metal, and oxide residue were then separately analyzed. Cuprite was calculated from the weight of copper found in solution after allowance had been made for malachite. Tables 1 to 15 list the results of analysis of twenty samples, and account for most of the material present. Some of the metallic samples showed oxide inclusions on metallographic examination; others revealed a spongy texture, suggesting that corrosion had penetrated deeply and left a network of metal and oxides. In such cases the analyses are incomplete because oxygen was not determined. The assumption that malachite is a pure compound ($CuCO_3 \cdot Cu(OH)_2$) is evidently reasonably correct, since the analyses total approximately 100 per cent even when this substance is an appreciable fraction of the whole sample.

Two samples belonging to phase G show that the melting of copper, and probably its reduction from an ore, was practiced at that time. Sample X-3024 consists of fragments of a clay vessel which was evidently used for melting (or smelting) copper. The analysis of the convex (exterior) surface (Table 1) establishes the material to be a typical clay. Adhering to the concave (inner) surface was a slag containing cuprite and malachite. Spectrochemical analysis of the malachite shows the presence of about 0.5 per cent arsenic, 1 per

[3] HILLEBRAND, W. F., AND G. E. F. LUNDELL, "Applied Inorganic Analysis," John Wiley & Sons, New York, 1929, p. 623.

[4] Metallic copper is virtually unattacked by hydrochloric acid if oxygen is excluded.

cent nickel, and 5 per cent tin. The second sample (X-3100) consisted of fragments of charcoal with adhering rock soil material and malachite. Table 2 gives the analysis of the pulverized charcoal. Fragments of the copper mineral separately analyzed gave 64.6 per cent copper. Malachite should contain 57.5 per cent copper, while cuprite contains 89.5 per cent copper. Spectroscopic examination of the pulverized charcoal showed the presence of both arsenic and nickel. It thus appears that ore reduction was practiced at the time corresponding to phase G, and the presence of a high concentration of tin in the charcoal suggests deliberate alloying. The possibility of co-reduction of a mixture of oxides is not excluded, however.

It seems likely that arsenic and nickel were not deliberately introduced, since their presence would serve little useful purpose as compared with tin. They were probably present in the copper ore, but proof of this cannot be given until ore or native metal from the Near East is found and analyzed. The presence of as much as 5 to 10 per cent of tin indicates a knowledge of bronze-making and of the fact that tin improves the casting qualities of copper. Tin is found in large concentration in such cast objects as the knob-headed pin (Table 4), the ax head (Table 12 and Figure 4), and the figurines (Table 9 and Figure 5), while with few exceptions those objects which were fabricated by forging (reamers and pointed pins) contain little or no tin. It seems reasonable to believe that there was some remelting of metal, which may account for the presence of tin in a few forged objects where it would not be expected.

In a number of cases the compositions of the metal and its corrosion products differ appreciably. Nickel and arsenic are invariably present to a lesser extent in the weathered material than in the metal, while there are indications that tin may be preferentially corroded (probably by local galvanic cell action). Some caution is evidently indicated in attempts to reconstruct the original metal composition from the analysis of corroded artifacts.

TABLE 8
Analysis of Long Pin (X-3316), Amouq Phase G

Constituent	*Core*	*Crust*	*Composite*
% of total	50.54	49.46	100.00
Cu	98.19	...	49.63
Pb	0.12	...	0.06
Ni	0.83	...	0.42
Fe	0.59	...	0.30
SiO_2	...	0.45	0.22
Insol. oxides	...	0.71	0.35
Malachite	...	7.53	3.72
Cuprite	...	87.67	43.36
Moisture, 110°C.	...	2.60	1.29
Ign. loss, 700°C.	...	5.80	2.87
	99.73	104.76	102.22

TABLE 9
Analysis of Bronze Figurines (X-5100), Phase G

Constituent	*Male*	*Female*	*Female (corrosion)*
Cu	76.0	84.3	74.1
Sn	7.4	10.9	15.0
Ni	...	0.26	...
Pb	0.0	0.09	0.0

TABLE 10
Analysis of Huge Pin Head (T-3340), Amouq Phase J

Constituent	*Percentage*
Cu	94.78
Sn	1.92
Fe	0.16
Ni	0.42
Pb	0.67
As	0.007
	97.96

TABLE 11
Analysis of Long Rolled-Head Pin (T-3373), Amouq Phase J

Constituent	*Core*	*Crust*	*Insoluble*	*Composite*
% of total	96.06	3.82	0.12	100.00
Cu	95.16	...	...	91.41
Fe	0.46	0.10	...	0.45
Ni	0.49	0.10	...	0.51
As	0.87	0.03	...	0.85
SiO_2	...	...	33.7	0.04
Insol. oxides	...	...	66.3	0.08
Malachite	...	13.33	...	0.51
Cuprite	...	87.77	...	3.35
	96.98	101.33	100.0	97.20

Figure 5. Male and Female Figurines, Amouq Phase G

TABLE 12
Analysis of Axe Head (T-3374), Amouq Phase J

Element	*Percentage*
Cu	88.37
Sn	9.74
Pb	0.30
Fe	0.07
Ni	0.17
As	0.032[a]
	98.68

[a] Colorimetric determination; 0.031% As by gravimetric method.

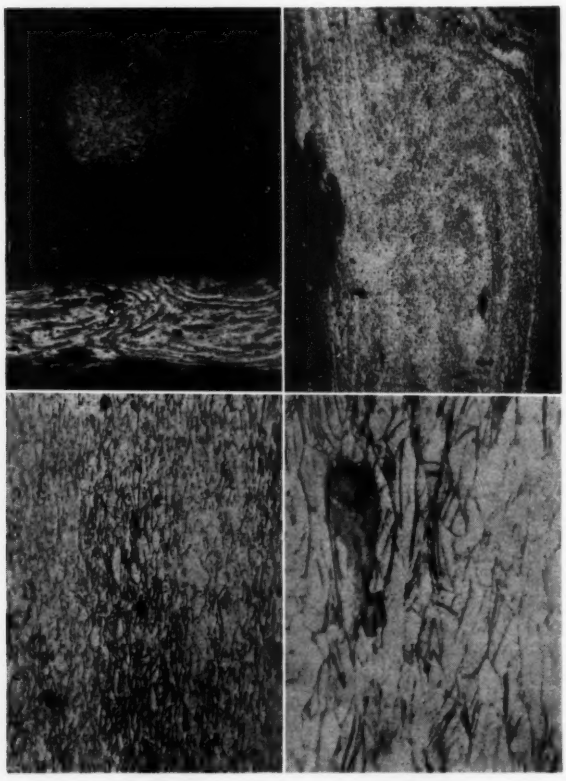

Figure 6. Photomicrographs of Polished Sections of Reamer 1, First Mixed Range

TABLE 13
Analysis of Dome-Headed Toggle Pin (T-3544), Amouq Phase J

Constituent	*Core*	*Crust*	*Insoluble*	*Composite*
% of total	89.28	10.57	0.17	100.02
Cu	97.54	...	...	87.08
Fe	0.55	Trace	...	0.49
Ni	0.22	0.12	...	0.21
As	0.07	0.001	...	0.06
Insol. oxides	...	...	100.0	0.17
Malachite	...	7.53	...	0.80
Cuprite	...	70.79	...	7.48
	98.38	89.01	100.0	96.31

METALLOGRAPHIC EXAMINATION

Metallographic examination is a powerful aid in deducing the history of a piece of metal since it permits direct observation of the internal structure of the piece, and almost every mechanical or thermal treatment causes changes in structure. If the metal were cast to the present shape, the individual crystals or grains of which the metal is composed are frequently elongated in the direction of heat flow during solidification. In addition, if the metal is an alloy (and all except the purest metals must be considered to be alloys) then by a phenomenon called coring, segregation of the various constituents may occur during solidification, giving rise to a "dendritic" structure—a pattern of branching lines in each grain. These characteristic microstructures enable an "as-cast" specimen to be readily identified. If the metal is worked, the grains, coring lines, and any inclusions of slag or other dirt are drawn out in the working direction so that the flow pattern can be observed. If the metal is worked hot, or is annealed after working, recrystallization occurs, and a new generation of crystalline grains, smaller than their "as-cast" parents, appears. One can be sure that recrystallization occurred if the grains present are considerably smaller than the spacing between the coring lines. Freshly recrystallized grains are roughly equidimensional polygons; if, however, the specimen is worked below the temperature at which recrystallization occurs, the grains will be drawn out in the direction of working. Thus an estimate of the upper limit of the final working temperature can be made from an observation of grain shape. The conclusions which are presented here concerning possible methods of fabrication were deduced from observations of this general type.

A photograph of the six reamers is shown in Figure 3, and various data concerning them are presented in Table 3. Longitudinal and transverse sections of Specimen 1 from the First Mixed Range are shown in Figure 6*a* at 20 diameters. The dark etching bands arranged like the grain in wood are coring lines which result from segregation during solidification. Had the specimen been worked down smoothly from the original ingot, these lines would have been parallel, and their confused pattern indicates a lack of skillfulness on the part of the fabricator. In the transverse section, the flow lines show how the surface of the specimen was actually folded back upon itself during hammering. This is a flaw which is by no means rare even in modern metals, but in this group it was observed only in this and, to a lesser extent, in the following specimen. The magnification in Figure 6*c* is sufficient to reveal the individual grains and shows that they are much smaller than the spacing between the cored dendrites and are elongated in the direction of working. It is clear then that the piece was heated during fabrication to produce the very small grains and that it was worked down by a large amount at a relatively low temperature after the final heating to produce the elongated grains. One would estimate that the cross-sectional area of the piece must have been reduced by at least one-third to account

for the marked elongation of the grains now present.

Several photomicrographs of Specimen 2, from phase F, are shown in Figure 7. The coring in this specimen is slight, but flow lines are still visible in Figure 7*a*, for example. Their waviness again indicates rather unskillful fabrication. The numerous rather large slag inclusions, which can be readily seen in the photomicrographs, have frequently acted as centers for corrosion. In several places the surface has been folded in by working, and some corrosion has also occurred at these sites. The small grain size indicates that the metal was worked hot or annealed during working, but the fact that the grains are equi-axed shows that no working was done subsequent to the last heating, or at least after the specimen had cooled below the recrystallization temperature.

Specimen 3, from phase G, appears to have been fabricated by a more experienced craftsman than the earlier reamers. It is true that the metal shows more inclusions than specimen 1, but the parallel flow lines which are clear in Figure 8*a* suggest that the piece was deftly beaten into shape. The coring lines are closer together than in the previous specimens, which might mean that the original ingot was larger or could, of course, merely mean that the "as-cast" grain size in the ingot was smaller. Straight sided bands can be seen in the grains in Figure 8*c*; these are annealing twins which appear only in metals that have been worked and heated. The final grains are somewhat elongated, showing that some shaping was done below the temperature at which recrystallization could occur; possibly the specimen was worked hot, and the finishing touches applied after it had cooled below the recrystallization temperature.

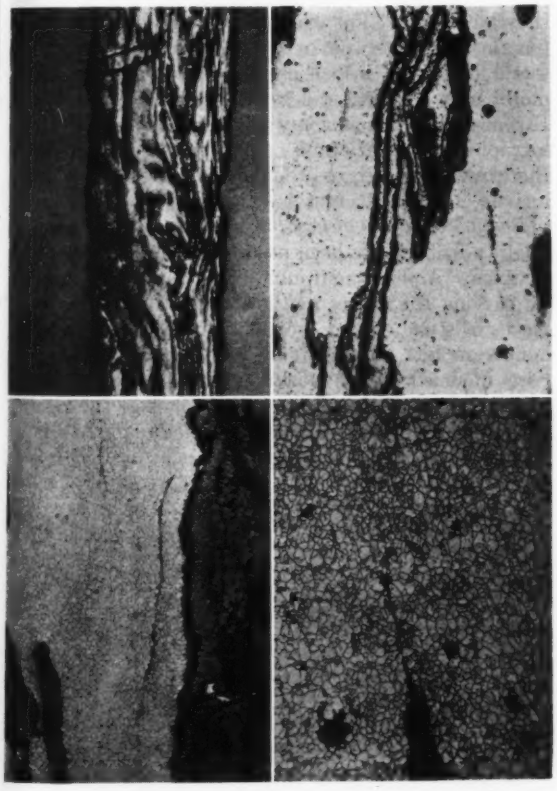

Figure 7. Photomicrographs of Polished Sections of Reamer 2, Amouq Phase F

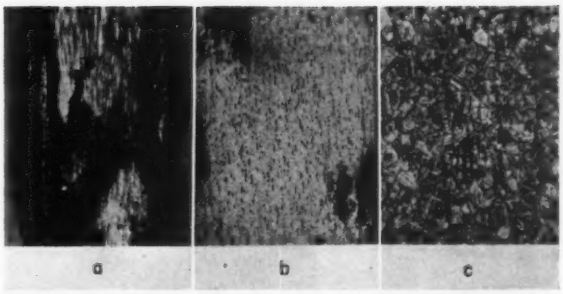

Figure 8. Photomicrographs of Polished Sections of Reamer 3, Amouq Phase G

Although Specimen 4, from phase H, suffered severe localized corrosion, as can be seen in Figure 9*a*, the other photographs in Figure 9 show that much good metal remains. The segregation during solidification was severe. The dark and light bands in Figure 9*d* are revealed by the differences in the rate of attack of regions of different chemical composition by the etching reagent. The parallelism of the flow lines indicates expert fabrication, and the closeness of their spacing may indicate a rather large ingot. Again, one con-

TABLE 14

Analysis of Long Rolled-Head Pin (T-3599), Amouq Phase J

Constituent	*Core*	*Crust*	*Insoluble*	*Composite*
% of total	97.71	2.26	0.12	100.09
Cu	98.01	...	...	95.77
Fe	0.37	0.11	...	0.36
Ni	0.29	Trace	...	0.28
As	0.04	0.008	...	0.04
SiO_2	...	...	0.00	0.00
Insol. oxides	...	...	100.00	0.12
Malachite	...	0.00	...	0.00
Cuprite	...	96.17	...	2.17
	98.71	96.39	100.00	98.74

TABLE 15

Analysis of Dome-Headed Toggle Pin (T-3800), Amouq Phase J

Constituent	*Core*	*Crust*	*Insoluble*	*Composite*
% of total	94.74	5.26	0.00	100.00
Cu	95.68	...	...	90.65
Fe	0.17	0.83	...	0.20
Ni	0.58	Trace	...	0.55
As	0.032	0.015	...	0.03
Insol. oxides	...	...	...	0.00
Malachite	...	0.00	...	0.00
Cuprite	...	96.18	...	5.06
	96.46	97.02	...	96.49

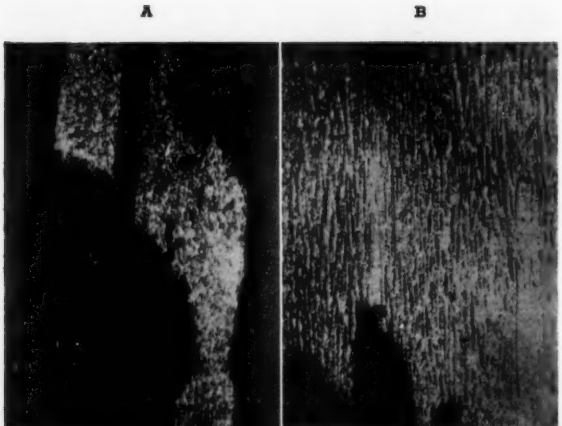

Figure 9. Photomicrographs of Polished Sections of Reamer 4, Amouq Phase H

cludes from the presence of annealing twins and a fine grain size that the metal was heated during or subsequent to working, and the slight elongation of the grains visible in Figure 9c shows that some final working was done below the recrystallization temperature.

While the workmanship displayed in the reamers from phases G and H was excellent, that in Specimen 5 from phase I is again poor. The metal is full of large slag inclusions indicating poor melting or casting practice, and the confused flow lines suggest that the workman had difficulty in producing the desired shape. Possibly by this time production of such simple objects was delegated to an apprentice. The structure shown in Figure 10c is difficult to interpret in terms of fabrication procedure. Metallurgically, it is a partially recrystallized metal; there are long bands of hard cold-worked metal separated by bands of soft, fine-grained recrystallized metal. Although this specimen has almost the same composition as Specimen 2, it is much harder because it has not been annealed to a temperature sufficiently high to completely recrystallize and soften it. The easiest way to duplicate this structure would be to cold work the metal heavily and then heat it for a short time at a temperature where recrystallization would proceed very slowly, but possibly it could be produced by hot working the specimen at a relatively low temperature. If so, the piece would have become progressively harder as working proceeded, and this might account for some of the apparent difficulty that was encountered in forming it.

Specimen 6, also from phase I, came from a different site and appears to have been made from an intentional tin alloy. It is well made and apparently was made with facility since the flow lines are parallel. The high hardness of Rockwell F 108 is largely due to the tin present. (See Figure 11.)

EXAMINATION OF BRONZE FIGURINES

The group consists of three male and three female figures, all of which were obviously cast, so this report is confined largely to speculation, based upon a detailed examination of the figures, as to the method of casting.

Two of the males and two of the females are similar in appearance and are larger than the remaining pair, although more crudely modeled in the flat. The small pair are modeled in the round and represent in most respects more expert craftsmanship. One large male and one large female are rather badly corroded, but the other four pieces are excellently preserved (Figure 5).

Metallographic Examination. One can conclude immediately from the appearance of the specimens that they were cast. For example, in casting some metal remains in the sprue or opening through which the metal is poured into the mold; the remains of this metal may be seen under the feet of all the figures. However, this conclusion was confirmed by the metallographic examination of a small piece cut from one of the sprues which shows the dendritic structure characteristic of "as-cast" metals (Figure 12).

Possible Methods of Casting. At least three methods might possibly have been used to prepare molds for the castings. A permanent mold could have been prepared by carving the pieces in the negative in a number of pieces of stone or clay, so they would assemble into a hollow mold. Alternatively, the figures could have been modeled in the positive, and then coated with moist clay after greasing. After drying, the coating could be cut into as many pieces as necessary to remove it, and these reassembled to give the desired hollow

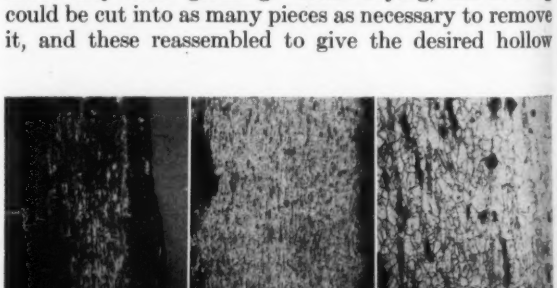

Figure 10. Photomicrographs of Polished Sections of Reamer 5, Amouq Phase I

mold. This method is often used at the present time in the casting of statuary. The third possible method is the lost wax process which in more recent years has been called investment casting. The process is known to be older in Mesopotamia than in Egypt.[5] It was also used during the recent war for the fabrication of parts of high melting, non-machinable metals for turbo-superchargers and jet engines. The procedure is simple. A model of the final part is prepared in a suitable wax, and this is then invested or coated with a slurry of a refractory material such as clay. After drying, the invested wax is coated with more clay, or packed in sand, and the whole assembly heated to melt out the wax and leave the mold ready to receive metal.

Of these methods, the first can be ruled out immediately. The appearance of the pieces is definitely that of figures modeled in the positive in some soft material like clay. The decision must thus be made between the second and third of the methods described above. If the mold had been made by coating a positive model with clay and cutting it into small pieces to remove it from the pattern, a very large number of pieces would have been required, since the shape is complicated and difficult to draw from a mold. There are numerous reasons for rejecting this possibility, but the most important is that after reassembly into a completed mold there would have been numerous small cracks into which metals would flow to produce fins on the finished casting. A careful search for these has been made, and no evidence of them can be found. Either they were never present or they were carefully removed in a final finishing operation. A few very obvious imperfections were found, however: for example, a ridge on the left thigh of the large male and two large lumps and a long ridge on the back of the same male. Most of these could have been easily removed if a finishing operation had been performed. Since no attempt was made to remove these obvious imperfections, one is left with the conclusion that no finishing was done on the rough casting and thus that this second method of making molds was not used.

A comparison of the two large females gives further support to the conclusion that these figures could not have been cast in a permanent mold or in a mold that was mechanically produced from a master pattern. Although the general appearance of the females is similar, they differ sufficiently in important details for it to be impossible that they are mechanical reproductions of the same master pattern. For example, while the hips are broader in one of the pair, the shoulders are broader in the other, and a comparison of the carving on the backs shows that this also differs seriously from one to the other.

There is no doubt but that the figures were made by the lost wax casting process, with the individual patterns probably being beeswax models of standard figures and the investment material probably clay. This procedure would give highly perfect castings which would reproduce fine detail in the original and which would require little, if any, finishing. The imperfections previously noted undoubtedly came from cracks in the mold which might have appeared either when the wax was melted out or when the hot metal was poured in.

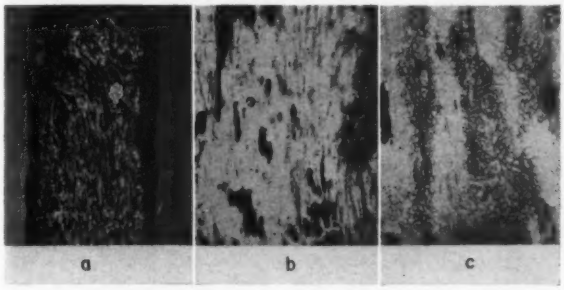

Figure 11. Photomicrographs of Polished Sections of Reamer 6, Amouq Phase I

It is evident that all the figures were cast in the inverted position. This is shown both by the position of the sprue under the feet and of porosity at and under the chins of almost all the specimens; in an inverted specimen the gas, which causes the porosity, would, of course, collect at a re-entrant angle such as exists at the chin. It is interesting to note that in the tall figures which have long feet, the sprue was arranged to run from front to back, to feed the feet properly with molten metal, while in the small specimens with short feet, it was possible to orient the sprue in a left-right plane.

The method used to produce the various holes in the casting is uncertain. Very probably the holes in the hands were cored in the mold, since they are rather large. It is not impossible that the holes around the head, through which the wires which were used to repre-

[5] In Egypt, the older claims of H. GARLAND, AND C. O. BANNISTER, "Ancient Egyptian Metallurgy," **1926,** p. 36, that the VIth Dynasty statue of Pepi (*ca.* 2300 B.C.) were so cast, have now been treated with skepticism by J. R. PARTINGTON, "Origins and Development of Applied Chemistry," **1935,** p. 55, 64. Partington does not believe the use of the lost wax process for bronze casting came into vogue in Egypt before the XVIIIth Dynasty, although its use for jewelry casting in the finer metals may go back to the XIIth Dynasty. Dr. Cornelius Hillen, of the Oriental Institute, who gave us very helpful advice in these matters, believes that the earliest possible indications of such casting may be found in the small copper animal which forms a handle on a cylinder seal of the Proto-Literate *b* phase (late "Uruk period") in Mesopotamia; see H. FRANKFORT, "Cylinder Seals," **1939,** pl. I-b. A date for this cylinder seal would be about 3400 B.C. The lost wax process is considered to have been certainly used in the production of a group of three copper statuettes found by the Oriental Institute at Khafajah, in Iraq, in Early Dynastic II context (*ca.* 2800 B.C.); *cf.* P. DELOUGAZ, "The Temple Oval at Khafajah," O.I.P. **53,** (1940), figs. 28, 29, and p. 151; H. FRANKFORT, "Sculpture of the Third Millenium B.C. from Tell Asmar and Khafajah," O.I.P. **44** (1939), p. 39 and pls. 98–103; and H. FRANKFORT, "More Sculpture from the Diyala Region," O.I.P. **60** (1943), p. 11. The Khafajah statuettes are probably slightly later in time than are the figurines from the Amouq phase G under discussion here.

sent hair were later passed, were cored in the mold, but this would have been difficult. It seems more probable that they were drilled in the finished casting.

CONCLUSIONS

A fairly good picture of metallurgical knowledge, and of its development over the period represented by these specimens, can be assembled from the data presented here. Although some of it must be regarded as speculative, most of it is well established.

The copper-nickel-arsenic alloys are probably natural alloys, since both nickel and arsenic are common impurities in copper ores. Nevertheless, the arsenic would act as a deoxidizer and would facilitate the casting of reasonably sound ingots for forging. Tin, on the other hand, rarely if ever occurs with copper, and it seems

Figure 12. Polished Section of Statue

Shows dendritic structure characteristic of cast metal.

almost certain that the rather large amounts found in artifacts from phase G and later were intentionally added. The fact that no tin-bearing metals were found prior to phase G may mean that its advantages were not discovered until that time. On the other hand, the fact that the use of the bronzes was confined to castings may indicate not only that its advantages for casting were appreciated, but also that tin was scarce. Thus, if more early specimens were available, evidence for its earlier use might be found.

It is certain that metal *melting*, at least, was practiced in phase F, because the microstructure of the reamer from that phase, as well as from the First Mixed Range, indicates that the reamer had originally been cast as an ingot not many times larger than the final piece. The charcoal and crucible from phase G give definite evidence that smelting, that is, reduction of the ore with carbon, was practiced by that time and also that tin was added, possibly by co-reduction of the oxide.

All the forged reamers were recrystallized, indicating that they were either annealed at some stage during working, or that they were worked hot. Although no choice can be made between these possibilities from microstructural evidence, either one implies a good knowledge of the properties of metals. Most of the reamers had been cold worked to some extent; this hardens the metal, but since none of them was extensively cold worked, it would be improper to conclude that the object of the cold working was to increase the strength of the metal.

The six figurines from phase G are beautiful examples of lost wax castings and undoubtedly some of the simpler shapes were cast in permanent molds.

The amazing thing about all of these specimens is that the full-blown development of metallurgical art seems almost to coincide with the appearance of the first metal specimens. This cannot necessarily be taken as a historically valid generalization; however, the archeologist must always be bound to respect the possible accidents of deposition and reclamation. The areas exposed by the Syrian Expedition in the depths below phase G were restricted in size, and there is no proof that metal objects might not occur (although perhaps in relatively small numbers) if larger exposures were made in the earlier phases. The earliest reamers examined showed that either annealing or hot working and metal melting were known and, by phase G, smelting; copper was alloyed with tin, and lost-wax casting of high perfection was used. The processes or techniques do not differ except in scale from those now in use. More very early specimens must be examined to determine whether this indicates a rapid local development of technology, or whether the art was introduced from other areas.

Ancient Syria had a peculiarly important role as a crossroads for influences from the two great centers of early historical civilization in Egypt and Mesopotamia, following 3000 B.C. Its advent into the earliest village stage probably came about at a time prior to the beginning of that stage in either classic Egypt or Mesopotamia—it lay within the hilly flanks of the "fertile crescent" (Figure 1) where agriculture and the village-peasant economy seem to have begun—somewhere towards 5000 B.C. It can be maintained that the economic foundations and the beginnings of the technological knowledge which made Western civilization possible were laid in these two thousand years and in this general area. This paper is, if nothing else, a demonstration of how the archeologist and the physical scientist may work toward a fuller understanding of this crucial period in our cultural history.

SODIUM-FUSION TESTS ON PLASTICS

HOWARD NECHAMKIN
Pratt Institute, Brooklyn, New York

THE sodium-fusion test, as it is normally run in laboratory sections of organic chemistry, is quite standardized. A mixture or substance of undisclosed composition is handed to the student, who, in turn, subjects the material to the familiar routine of elementary analysis: fusion with sodium, leaching with water, and testing for elements now present as halide, phosphate, sulfide, cyanide, etc. It has been my experience that this experiment, although useful beyond measure, usually fails to elicit the active interest of the class. It appears that simply "testing" for the various elements present is not an end in itself, but a means to an end—and the students realize that.

In an attempt to make the tests more real for the learner, one technique which has been employed is to state that the "unknown" may be one of several compounds, *i. e.*, urea, thiourea, sucrose, sulfanilamide, *p*-dichlorobenzene, etc., and identification of the elements present will serve to make the choice among the various possibilities. This depends, of course, upon selection of such a group that any elements tested for appear in any combination in only one of the compounds suggested. This approach met with favorable response and was manifested by greater interest, on the part of the students, in the results of their work.

It was decided to pursue this line of approach a step further into the realm of reality by choosing as unknowns materials with which students would be quite familiar, the identification of which would provide even more benefit. This led to the selection of the ever-interesting group of plastics products. Unknowns in the form of bulk and powdered commercial plastics were distributed and, on the basis of the sodium-fusion test and a few added simple tests, experiments were made to identify them.

A list of possible products was posted in the laboratory, together with pertinent information regarding their behavior, and the students were expected to identify the particular plastics given them.

First Separation into Groups by Fusion

I. Specimens giving a positive halogen test and no other:
   - A. Polyvinyl chloride or mixed chloride-acetate (Vinylites)
   - B. Vinylidene chloride (Saran, Velon)

II. Specimens giving a positive nitrogen test and no other:
   - A. Cellulose nitrate (Celluloid)
   - B. Urea formaldehyde
   - C. Polyamide (Nylon)

III. Specimens giving a positive sulfur test and possibly halogen:
   - A. Polysulfides (Thiokol)

IV. Specimens giving positive tests for nitrogen and sulfur:
   - A. Thiourea-formaldehyde

V. Specimens giving positive tests for nitrogen, sulfur, and phosphorus:
   - A. Casein

VI. Specimens apparently containing only carbon, hydrogen, and possibly oxygen:
   - A. Polystyrene (Styron)
   - B. Phenol-formaldehyde
   - C. Cellulose (Cellophane)
   - D. Cellulose acetate (Lustron)
   - E. Acrylate resins (Lucite)

Distinction Among Group Members

I. A. Vinyl chloride or acetate copolymer resins are generally soluble, although slowly, in acetone and toluene. When held in a flame they soften and melt rapidly during their decomposition.
   B. Vinylidene chloride resins are generally resistant to the solvent action of acetone and toluene. They usually are prepared for commercial use as filaments or tubing. When held in a flame, they decompose without appreciable melting and exhibit a heavy ash.

II. A. Cellulose nitrate plastics burn vigorously, when ignited, with a bright flame.
   B. Urea formaldehyde resin is flame resistant. It will not burn readily and will only char or discolor when heated. It does not melt.
   C. Polyamides, when heated, melt and then burn with some difficulty.

III. Needs no further distinction.

IV. Needs no further distinction.

V. Needs no further distinction.

VI. A. Polystyrene resins will burn slowly, when ignited, with a smoky flame. They are soluble in aromatic hydrocarbons and chlorinated solvents, but not in ketones.
   B. Phenol-formaldehyde follows the behavior of urea resins (II, above).
   C. Cellulose is the regenerated and extruded (usually) material. It burns like paper and is not soluble in organic solvents. It is usually colorless or transparent.
   D. Cellulose acetate resin takes fire with difficulty and usually sputters until self-extinguished. It is readily soluble in acetone.
   E. Acrylate resins burn slowly, when ignited, with a predominately blue flame. They are

soluble in aromatic hydrocarbons and in such ketones as methyl ethyl ketone.

On the basis of these simple tests, *i. e.*, sodium fusion, burning behavior, solubility characteristics, etc., the student is able to classify his substance. The thirteen types of plastics here employed are characteristically those which are most widely used for commercial and industrial purposes. In general, each of the types is represented by many different trade names on the market and it should be pointed out, for instance, that the names Aceloid, Celluloid, Celluvarno, Herculoid, Hycoloid, Kodaloid, Lusteroid, Nitron, Plastini, Samson, and Simco are all examples of cellulose nitrate plastics and that such classification greatly simplifies this vast group of commercially important products.

In distributing specimens to the class the same type of plastic must be given to more than one student. This difficulty is solved by using different colors or forms of the various materials. When notes are compared in the laboratory, as they always will be, even more may be learned by the group in observing the different outward appearance the plastics may assume depending on the mode of fabrication.

LABORATORY EXERCISES IN NUCLEAR CHEMISTRY

IV. A Basic Student Counting Circuit

WILLIAM H. HAMILL, RUSSEL R. WILLIAMS, JR., and COLIN F. MACKAY
University of Notre Dame, Notre Dame, Indiana

In previous articles of this series (*1*, *2*, *3*) we have described simple experiments in nuclear chemistry designed for use in undergraduate laboratory instruction and have listed (*2*) the essential equipment for this type of work together with approximate costs. Excepting the 5-mg. Ra-Be neutron source, the most essential and expensive item is the electronic circuit for operation of a Geiger tube. Those available commercially fall into two classes: (1) rate meters, which give an indication of the average count rate over a fixed period of time, and (2) scaling circuits, which total the number of counts in any period of time. Items of type (1) may be purchased for as little as $100 but are only semiquantitative in operation and are intended chiefly for demonstration purposes. Type (2), the scaling circuits, are excellent for quantitative work but cost $300 or more.

For an institution with a research program in nuclear chemistry, or able to purchase a scaling circuit for instruction, the choice is clear. However, when funds are limited or when multiple instructional units are desired, the rate meter type is much less expensive but also less satisfactory. Aside from the inherently qualitative nature of the inexpensive rate meter, we find that it lacks certain desirable pedagogical advantages of the scaler, namely, direct indication of the counts received and quantitative response at low counting rates.

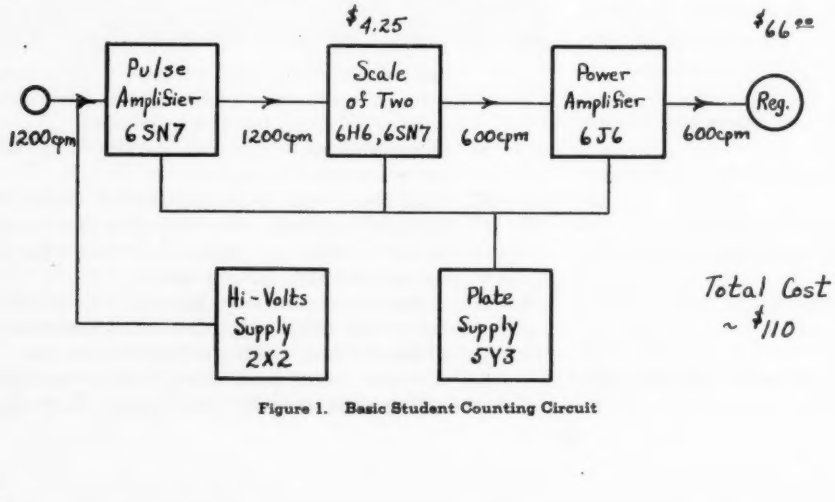

Figure 1. Basic Student Counting Circuit

With these considerations in mind we have set out to explore the possibility of constructing an adequate inexpensive student counter circuit. This work has resulted in the completion of one such circuit at a cost in parts of approximately $110, described below, and the realization that further simple modification can re-

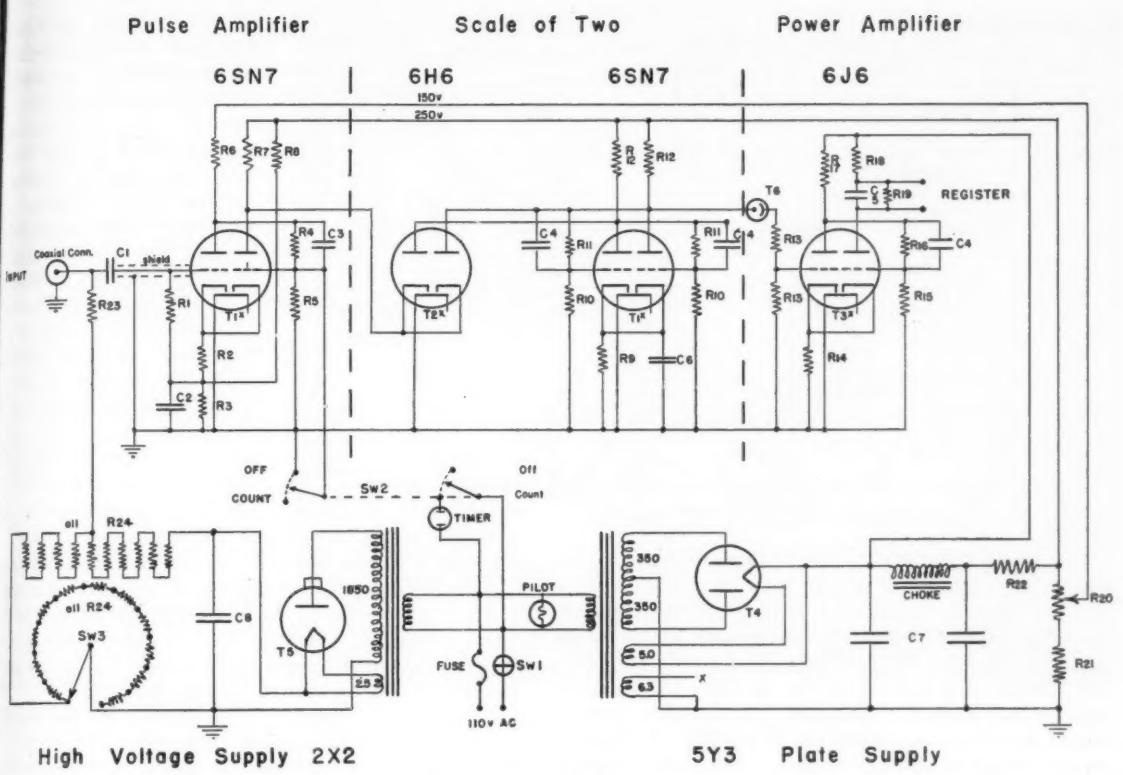

Figure 2. Student Counter Circuit

Table of Parts (9)

	Resistances		*Resistances (cont.)*		*Miscellaneous*	
R1	150K, 1/2w	R22	1K, 5w		Pilot light assembly (110 v.)	
R2	100K, 1w	R23	1 meg., 1/2w		Fuse assembly	
R3	2.7K, 2w	R24	250K, 1/2w	$7.50	Switch Sw-1, SPST (on-off)	
R4	400K, 1/2w				Switch Sw-2, DPDT (count-stop)	
R5	100K, 1/2w		*Capacitors*		Switch Sw-3, 10–12 pos. (voltage control)	
R6	50K, 1w	C1	50 μμfd, 2000 v.		Tube sockets (octal)—5	
R7	1.5K, 1w	C2	25 μfd, 25 v.		Tube socket (4-prong)—1	
R8	25K, 1w	C3	500 μμfd, 300 v.		Tube cap connector	
R9	6.8K, 2w	C4 (3)	50 μμfd, 300 v.		Two prong female connector (timer outlet)	
R10 (2)	100K, 1w	C5	0.05 μfd, 300 v.		Coaxial connector (male and female chasis mount)	
R11 (2)	200K, 1w	C6	0.1 μfd, 50 v.		NE51 pilot lamp	
R12 (2)	20K, 1w	C7	10-10 μfd, 350 v.		Dial plate	$ 5.84
R13 (2)	5 meg., 1/2w	C8	0.5 μfd, 2000 v.	$8.53		
R14	5K, 2w				Transformer, Stancor P6011	4.53
R15	100K, 1/2w		*Tubes*		Transformer, Thordarson T22R42	4.26
R16	250K, 1/2w	T1 (2)	6SN7		Choke, 8–10 henry	1.47
R17	10K, 2w	T2	6H6		Chassis and cabinet	6.57
R18	5K, 2w	T3	6J6		Total cost as shown in Figure 2 exclusive of register	$46.36
R19	100K, 1/2w	T4	5Y3		Cyclotron specialties register, type 401B (4)	$66.00
R20	5K, 10w VT	T5	2X2		"Mercury" register (5)	$ 4.00
R21	5K, 10w	T6	Ne2	$7.66	Additional scaling stages, each approx.	$ 4.35

duce the cost of parts for such a unit to as little as $65. All parts used are readily available, and no great skill is needed in the construction.

COUNTING CIRCUITS

The maximum counting rate which need be encountered in student experiments lies in the region 1000–2000 counts per minute. At this rate a statistically significant count can be collected in a few minutes. Although some electro-mechanical registers will operate at such a rate on evenly spaced impulses, the random pulses obtained from nuclear events may frequently occur at such small intervals as to be indistinguishable to such a register. This loss is commonly reduced by

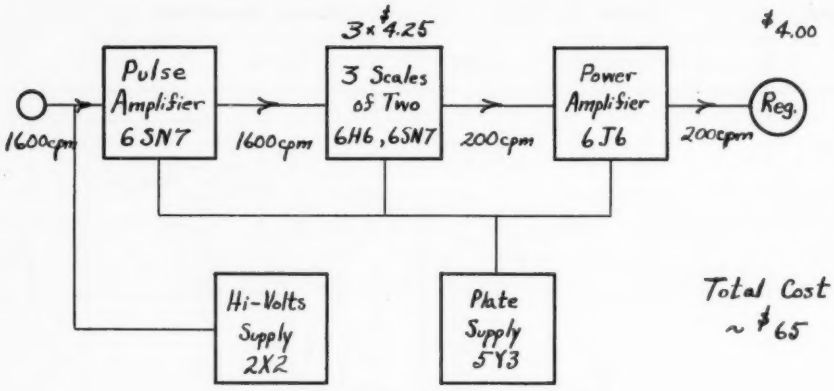

Figure 3. Modified Student Counting Circuit

use of an electronic scaling circuit which reduces the count rate delivered to the register to some acceptable value.

Scale-of-two and Fast Register. One acceptable combination of scaling circuit and register is the use of a very fast electro-mechanical register (such as Cyclotron Specialties No. 401-B (*4*)) and one scale-of-two, as shown in the block diagram of Figure 1. The register will operate at a maximum rate of 100 evenly spaced impulses per second. Therefore, the loss with random counts should be approximately 10 per cent at a rate of 10 per second or 600 per minute. With one scale-of-two, this will permit counting at rates up to at least 1200 counts per minute, with loss of less than 10 per cent. The actual loss is less than this, due to the regularizing action of a scaling unit as discussed below, and in any event the loss can be determined (*2*). This combination of one scale-of-two with a fast register is not the least expensive one but is electronically the simplest. The circuit is shown in Figure 2, with the necessary auxiliary components described below.

Scale-of-eight and Slow Register. A second acceptable and less expensive combination of components is represented by the use of three scales-of-two with a "slow" electro-mechanical register, such as the "Mercury" Register (*5*), as shown in the block diagram of Figure 3. The mechanical register in this case has a limiting rate of 10 evenly spaced impulses per second and therefore a loss of 10 per cent at one random pulse per second. Through a scale-of-eight, this would correspond to 8 counts per second or 480 counts per minute. However, the "regularizing" action of a scale-of-eight is considerable, and according to the treatment of Rainwater and Wu (*6*) a rate as high as 2000 counts per minute could be tolerated with less than 10 per cent loss in this arrangement. The construction of this circuit involves only a repetition of the scale-of-two as shown in Figure 2. All other components remain unchanged. This modification could be constructed for approximately $65 in parts.

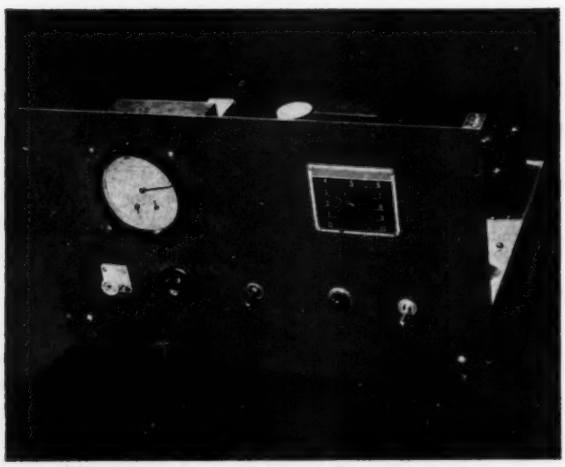

Figure 4. Student Counter (Front View)

High Voltage Supply. This portion of the circuit consists of the usual half-wave rectifier (2X2) driven by a transformer originally designed for a photoflash unit. The high-voltage, low-current-drain characteristic of such transformers is applicable here, and several other inexpensive transformers of this type might be used. A simple condenser filter is used here, with no other voltage regulation. Modern Geiger tubes have such broad and flat plateaus that electronic regulation is not essential. The control of high voltage is accomplished through a variation in the current drain, which permits minimum voltage across the voltage control. This is arranged to vary the output voltage between approximately 700 and 1200 volts in 50-volt steps. A meter to measure high voltage is considered an unnecessary expense. The high voltage circuit must be properly insulated to prevent breakdown, as illustrated by the use of a chain of resistors instead of a single unit.

Plate Supply. A 5Y3 full-wave rectifier feeds into a standard filter circuit. The various plate voltages desired are drawn at separate points in this circuit as indicated. Values given are approximate.

Pulse Amplifier. This portion of the circuit is a univibrator which is tripped by the negative pulse delivered from the Geiger tube through condenser C1. It delivers a large negative pulse at the second plate of the 6SN7 tube each time a discharge occurs in the Geiger tube. This circuit is very similar to that used by the Manhattan Project in their widely used scaling circuit.

Scaling Circuit. The well-known Higinbotham scaling circuit (*7*) is used here for two purposes: first,

to reduce the count rate by a factor of two and thus increase the maximum useful counting rate; and second, to deliver a large and long actuating signal to the power amplifier.

A scale-of-two has two stable states and is changed from one to the other by the negative trigger pulse from the preceding stage. The two halves of the 6SN7 should be quite symmetrical with respect to transconductance and the values of the resistors used. The output at the second plate will be a square wave whose frequency is set by the count rate.

Power Amplifier. This unit was developed in the Notre Dame Physics Department (*8*) and has been highly successful in driving many types of mechanical registers at high speeds. Its action follows that of the scale-of-two in that one count causes current to pass through the register coil, while the next count cuts off the current in the register coil. Thus the register records every other count, and its indication must be multiplied by two.

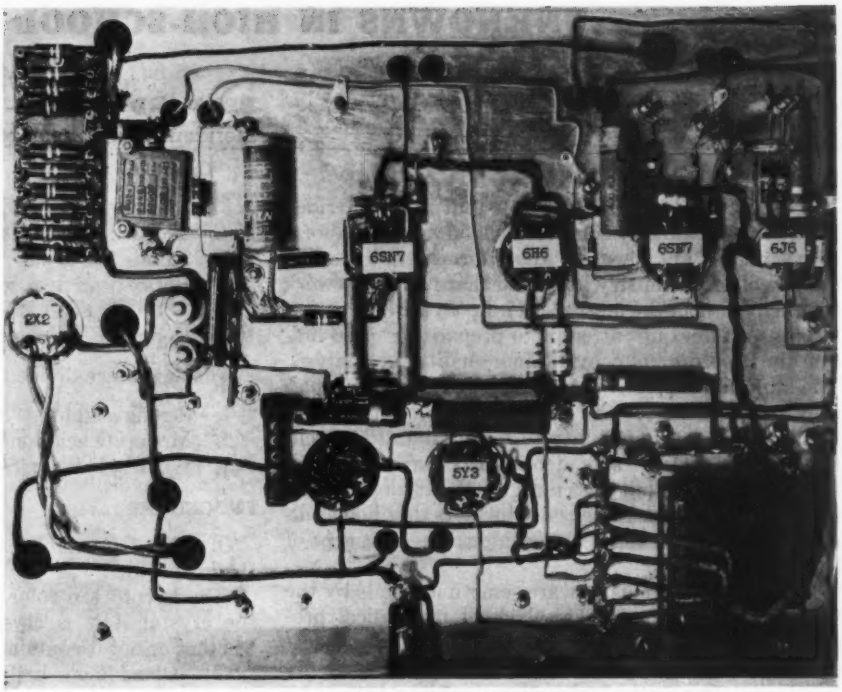

Figure 6. Student Counter (Wiring Details)

CONCLUSION

Detailed discussion of the design and operation of the various components is available elsewhere (*7*). However, the parts list supplied should permit construction of the circuit or the suggested modification by anyone with some slight acquaintance with wiring practice. The actual construction of this circuit was accomplished by one of us (C. F. M.) as a senior chemistry major with no previous experience with such circuits. Figures 4 and 5 show the general layout of parts, while Figure 6 is intended to aid in the wiring.

It is to be hoped that the low cost and relative ease of construction of the circuits described above will permit the adoption of nuclear chemistry experiments by a greater number of schools. Our convictions as to the importance and usefulness of some basic, widespread training in this field have been previously stated.

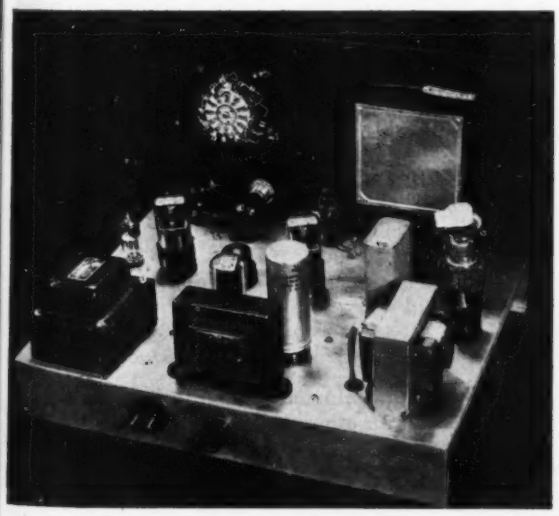

Figure 5. Student Counter (Back View)

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UNKNOWNS IN HIGH-SCHOOL CHEMISTRY

BERNARD JURALE
Meriden High School, Meriden, Connecticut

THE most enjoyable and valuable unit in the Meriden High School chemistry course is the two weeks of laboratory work on "unknowns." Many former students now taking college chemistry have said that it was the work that helped them the most in their first-year course.

This unit does not attempt to prepare for or to imitate the procedure of conventional qualitative analysis. The important objective is to teach facts and principles that are essential for the understanding of chemistry. It also offers an opportunity to teach laboratory techniques and to use facts determined experimentally. Laboratory work that does not give the student opportunity to base his next steps on the facts that he has already determined cannot be considered the best type of learning by doing.

Tests for the various ions are sometimes made by the "hit-or-miss" method. This lack of methodical procedure is wasteful of chemicals and time. Every test should be made with the thought of what has to be determined. The method of testing described here is an attempt to unify the accepted test procedures of high-school chemistry into a workable laboratory exercise.

We prefer to omit some laboratory work of doubtful value on the metals and to substitute a long period for topics that do not lend themselves to ordinary classroom procedures. We find that good laboratory techniques and use of the scientific method may be inculcated by a longer laboratory unit better than by an equal time spread out in weekly laboratory periods. Also, the work on the "unknowns" is an excellent opportunity to apply such topics as hydrolysis, electromotive series, solubility rules, and ionic reactions.

Hydrolysis is sometimes given inadequate consideration in high-school chemistry. This may be due to the lack of examples to show that hydrolysis plays an important part in chemical reactions involving ions. It is, however, a valuable asset in determining the relative behavior of the ions in a salt. Universal indicator paper gives a much better check on pH value of a salt solution than does litmus.

The preliminary work consists of reviewing the important ions and their identification. These tests have either been taken up previously in laboratory work or shown as demonstrations in the classroom. In order to coordinate the procedure the cations are classified according to method of testing and the anions by the strength of the related acids. Also, each student is assigned to a sample that is within his ability to identify.

The cation groups are as follows:

1. Ammonium ion.
2. Alkali metals: Li^+, K^+, Na^+.
3. Alkaline earths: Ba^{++}, Sr^{++}, Ca^{++}.
4. Cobalt nitrate test group: Mg^{++}, Al^{+++}, Zn^{++}.
5. Sulfide test group: Cd^{++}, Sb^{+++}, Pb^{++}, As^{+++}.
6. Borax-bead test group: Mn^{++}, Cr^{+++}, Co^{++}, Ni^{++}, Fe^{++}, Fe^{+++}.
7. Replacement group: Cu^{++}, Ag^+, Pb^{++}.

The anions are divided into three groups:

1. Strong acid ions: SO_4^{--}, NO_3^-, Cl^-.
2. Moderate acid ions: Br^-, I^-, CH_3COO^-.
3. Weak acid ions: SO_3^{--}, S^{--}, CO_3^{--}.

PROCEDURE

Obtain a 1-g. sample of a simple salt or an alum from the instructor.

(1) Record the color, appearance, and the form of the crystal if it is crystalline. Find out by a test whether or not it contains water of crystallization.

(2) Dissolve one-half of the sample in 25 ml. of distilled water. Test the solution with universal indicator paper and with litmus. Record the hydrolysis as *A* (strongly acid), *a* (weakly acid), *N* (neutral), *b* (weakly alkaline), *B* (strongly alkaline).

If the solution is not clear, determine whether the precipitate is caused by hydrolysis or by insolubility. In the former case, in what group would you expect to find the metal?

(3) Take a 3-ml. sample of the solution. Add sodium hydroxide solution carefully, a drop at a time, shaking after each addition. Record the appearance, color, and type of the precipitate, if any. If no precipitate, test for ammonium ion by warming and noticing the odor or the effect on moist red litmus held above the liquid in the tube.

(4) From the results of the hydrolysis test and from the color, type, or lack of precipitate, determine to what group the metallic ion is likely to belong.

	Precipitate	*Ions*
(*a*)	None	NH_4^+, Li^+, K^+, Na^+
(*b*)	White cloudy	Ca^{++}, Sr^{++}, Ba^{++}
(*c*)	White gelatinous	Mg^{++}, Al^{+++}, Zn^{++}
(*d*)	White heavy curd	Cd^{++}, Sb^{+++}, Pb^{++}
(*e*)	Colored	Ag^+, Fe^{++}, Fe^{+++}, Mn^{++}, Cr^{+++}, Co^{++}, Ni^{++}, Cu^{++}

(5) Group (*a*). Ammonium ion test given. Flame test for other three.

Group (*b*). Flame tests. Check Ca^{++} by lack of a precipitate when Calgon (sodium hexametaphosphate) solution is added before ammonium oxalate solution, and the

presence of a precipitate with ammonium oxalate solution in the absence of Calgon.

Or, to distinguish between Ca^{++} and Ba^{++} if the flame test is not conclusive, acidify the sample of salt to be tested with dilute nitric acid. Add, a drop at a time, a solution of dilute sulfuric acid. A precipitate will be formed by the addition of one drop of the acid if the salt contains Ba^{++} ions. Calcium salt will require much more acid to obtain a precipitate.

Group (*c*). Apply cobalt nitrate test.

Group (*d*). Add hydrogen sulfide solution or ammonium sulfide and observe color of precipitate.

Group (*e*). Mn^{++}, Cr^{+++}, Co^{++}, Ni^{++} are identified by the borax bead test.

Fe^{+++} gives a red color with KCSN solution. Alternatively, a blue color is obtained with $K_4Fe(CN)_6$ solution.

Fe^{++} is identified by a blue precipitate with $K_3Fe(CN)_6$ solution.

Cu^{++} will be replaced as a red metal by an iron nail, and Ag^{+} is replaced by a copper wire forming a black or a silvery deposit.

Flame tests can be made without the use of platinum wire. Ignite a splint and produce a carbonized section by putting out the flame with water. A small amount of salt will adhere to the charcoal. The flame test is made without interference from wood gases.

The borax bead test can also be made without platinum. Heat the end of a glass rod about 2 mm. in diameter. Thrust into powdered borax and reheat. Repeat until a drop of melted borax adheres to the rod. Pick up a small amount of sample and heat. Rotate the rod in the flame and the sample will mix evenly with borax. These beads may be saved for reference and comparison.

(6) When the cation has been identified, use the result of the hydrolysis test for an indication of the group to which the anion belongs. Make appropriate tests to identify the anion.

(7) Write equations for all the reactions used in identifying your compound and point out clearly the steps you used to reach the identification.

ACKNOWLEDGMENT

The writer wishes to acknowledge his indebtedness to Elbert Weaver for his help in revising this paper.

A METHOD OF DETERMINING THE THICKNESS OF SILVER DEPOSITED ON GLASS

LLOYD S. NELSON
Illinois Institute of Technology, Chicago 16, Illinois

THE formation of a film of metallic silver on glass is an experiment which illustrates, in general, the reaction of an aldehyde with an inorganic oxidizing agent, and in particular, the commercial manufacture of mirrors and the Tollens' test for aldehydes. Because it is of interest with respect to both the organic and inorganic compounds involved, this experiment is performed in both organic and general chemistry courses. An adequate description of the experimental details has been given elsewhere.[1]

As a companion experiment, the thickness of the silver film can be determined by a method described by Gardner and Case.[2] A small crystal of iodine is placed on the silvered surface (or, better, suspended about a millimeter above it) and shielded from air currents by an inverted beaker. Iodine vapor will react with the silver to form transparent silver iodide. As more and more of the silver is converted to the iodide, a transparent spot will appear. Around this spot, the silver will have been only partially converted into silver iodide, with the result that concentric, colored rings can be seen when the film is viewed by reflected light.

The number of rings present is a function of the thickness of the film. The accompanying table, calculated from the densities of silver and silver iodide and the refractive index of silver iodide, gives the relationship between the thickness and the number of rings.

Number of rings	*Thickness in millimeters* ($\times 10^6$)
2	31
3	61
4	92
5	123
6	154
7	215

With a knowledge of the thickness, it becomes possible to make a fairly accurate calculation of the amount of silver that was deposited. By comparing this figure with the original amount of silver, the "efficiency" of the process can be estimated.

[1] ARTHUR, P., "Lecture Demonstrations in General Chemistry," McGraw-Hill Book Company, New York, 1939, p. 370.

[2] GARDNER, I. C., AND F. A. CASE, Circular of the Bureau of Standards, No. 389 (1931).

THE ROLE OF THE LIBERAL ARTS COLLEGE IN THE PROFESSIONAL TRAINING OF CHEMISTS

HARRY F. LEWIS
The Institute of Paper Chemistry, Appleton, Wisconsin

THE Trytten "Report on the Baccalaureate Origins of the Science Doctorates Awarded in the United States, 1936–1945" lists the baccalaureate sources of those who were awarded earned doctoral degrees in the sciences (Ph.D., Sc.D., D. Eng., or D.P.H.) by institutions during the decade from January 1, 1936 to December 31, 1945, inclusive. This survey covered 28 scientific disciplines involving 14,905 doctor's degrees conferred by 90 institutions on graduates of 719 colleges and universities in the United States and 22 Canadian and 51 foreign universities.

The field of chemistry is represented by doctorates in chemistry, biochemistry, metallurgy, mineralogy, and pharmacology. Some 6182 doctorates in all were conferred in these five fields—with 5827 in biochemistry and chemistry alone.

Since the information in the Trytten report is relatively complete for the period, we would seem to have a means for evaluating the work of our undergraduate institutions as progenitors of our professional chemists, to the extent that these are represented by the Ph.D.'s and Sc.D.'s in chemistry. This is one measure of the over-all vitality of a chemistry department for a specific period.

The 719 institutions in the United States listed in the report were classified in four categories. The classification, together with the number in each category and the number of the degrees in *chemistry* and *biochemistry* in each category, are summarized in Table 1. (No attempt is made to classify those students who had their undergraduate training in Canadian and foreign institutions.)

TABLE 1
Summary of the Trytten Data by Institutional Classification

Type	*Number*	*% of total*	*Students to chemistry doctorate*	*% of total*
Universities of complex structure	144	20.0	3497	63.2
Colleges of arts and science	438	61.1	1694	30.6
Technical institutions	34	4.7	172	3.1
Teachers' colleges	83	11.5	147	2.7
No longer four-year schools	8	1.1	3	...
No longer functioning	12	1.6	18	...
Summary	719	...	5531	...

The data show that not quite two-thirds of the chemists receiving doctorates in the two fields noted have had their undergraduate training in the universities; almost one-third are trained in the arts and science colleges. Technical institutions and teachers' colleges account for less than 6 per cent of the total. Of the students earning doctorates in all fields of science listed in the Trytten report, 65.6 per cent are trained in the universities and 28.8 per cent in the colleges.

The undergraduate training of these chemistry doctorates was given in 567 of the above 719 institutions. Not a single student went out from 152 of the colleges and universities listed to complete the doctorate in chemistry; thus better than 21 per cent of our undergraduate institutions play no part whatever in the preparation for graduate training in chemistry. Of these, 24 are nationally accredited liberal arts colleges, 61 are regionally accredited colleges, and 19 are colleges which have received neither national nor regional accreditation. These 104 colleges make up about two-thirds of the nonproductive institutions; they also represent better than 23 per cent of the 438 colleges of arts and science listed in the Trytten report.

As a matter of general interest, when these figures are calculated back to the production of doctorates in terms of the ten-year registration of the average institution and the average doctorate production per institution, it would appear that universities and colleges on the average are equally productive. In an earlier paper[1] the writer has shown that the estimated average registration per institution for the ten-year period is 30,820 for the universities and 5000 for the colleges. When the number of potential chemistry doctorates trained in the universities in ten years is divided by the number of universities, we find that each institution trained an average of 24.3 for the ten years. For the colleges the figure is 3.9. Now dividing the average number trained by the average number of students registered—in the case of the universities 24.3/30,820—gives a value of 0.79 chemistry doctorate per 1000 registered. The analogous value is 0.78 for the colleges. Hence the statement that on the average they are equally productive.

The agency for rating work done by the various institutions in the matter of the professional training of chemists is the Committee on the Professional Training of Chemists of the American Chemical Society. Their Report 21 lists on pages 770 and 821 of *Chemical and Engineering News* for March 6, 1950, the schools on the approved list for the professional training of chemists. The list includes some 46 colleges of arts and science so listed in the Trytten report, together with 112 uni-

[1] Soon to appear in *The Journal of Higher Education.*

TABLE 2

Distribution of Chemistry Doctorates in A. C. S. Accredited and Unaccredited Institutions

Type	Number	—Undergraduate training in— Accredited	%	Accredited institutions	%	Not accredited	%
Universities of complex structure	144	112	77.7	3309	94.6	188	5.4
Colleges of arts and science	438	46	10.5	611	36.0	1083	63.9
Technical institutions	34	8	23.5	119	69.3	53	30.7
Teachers' colleges	83	0	...	...	...	147	100.0
Summary	699[a]	166	23.7	4039	73.3	1471	26.7

[a] This tabulation does not include the 20 institutions no longer four-year colleges or no longer functioning.

versities of complex structure and eight technical institutes. A few institutions accredited by the American Chemical Society do not appear on the Trytten report list.

The relationship between approval by the American Chemical Society in 1950 and the number of chemistry doctorates receiving their undergraduate training in approved and nonapproved institutions will be seen from the data in Table 2.

It is quite apparent that potential chemistry doctorates receiving their undergraduate training in universities do so almost entirely in schools approved for professional training by the American Chemical Society. Only slightly more than one in three taking their training in the colleges do so in institutions approved for professional training by that agency.

It is also quite apparent from a study of the records that there are a number of institutions having all the requirements for professional training (since they are on the approved list) which actually accomplished very little as this is measured by the achievement of the doctorate during the ten-year period covered by the report. Of the 46 colleges thus approved, the chemistry departments of some 19 have sent on the average through this period less than one to the doctorate per year; 11 of the 19 averaged one or less every other year; 8, three or less in ten years. Seven of these 19 colleges are women's colleges. By the same token, it is further quite apparent that there are a number of colleges not on the approved list whose chemistry departments have made a significant contribution to the undergraduate training of chemistry doctorates. One school in this category, Hope College, lists 24 of its undergraduates in the ten-year period who received the chemistry doctorate. Central of Missouri with 19, Wabash—18, Franklin & Marshall—17, Mississippi and Mt. Union—16, and Hiram—15 would all seem to have provided a favorable climate for predoctorate training.

One way to compare the activity of the various colleges would be to list them according to the number of the chemistry doctorates achieved by their graduates. This takes no account of the total number of graduates or the relative number of men and women. The top 22 have been so listed in Table 3.

A somewhat more realistic method of rating is achieved when we consider the number of doctorates in the light of the men graduates for the period which produced the majority of the 1936–45 doctorates—namely, the years 1932–41. This has been done. In order to screen out the most productive institutions, the following scheme was employed. The total number of B.A. and B.S. degrees awarded in the year 1938[2] was multiplied by ten, and this figure was assumed to represent the number of graduates of the various schools over ten years; the ratio of the number of doctors to the total number of graduates was then calculated as percentages, and schools showing a productivity of 1.5 per cent were investigated further.

Actual figures were then obtained from the majority of the schools on this list for the total number of graduates and for the number of men graduates for the years 1932–41, inclusive. The figures for the men graduates were used in calculating the percentage of chemistry Ph.D.'s per undergraduate school. This form of calculation would favor the coeducational schools to the extent that their women graduates earned chemistry Ph.D.'s. A random sampling indicated that this was not significant.

In Table 4 there have been arranged, in order of productivity, these colleges sending on a calculated 3 per cent or more of their men graduates in 1932–41, inclusive, to chemistry doctorates in 1936–45, inclusive.

These 14 colleges certainly qualify as outstanding in the stimulation of graduate work in chemistry during the years 1932–41. They are equaled on this same basis by not more than two universities—the California

[2] Taken from the "College Blue Book," 1939.

TABLE 3

Rating of Arts and Science Colleges by Number of Graduates Achieving the Chemistry Doctorate 1936–1945

Rating	School	Doctorates in biochemistry and chemistry
1	Oberlin	56
2	DePauw	44
3	Dartmouth	33
4	Brooklyn	30
5	St. Olaf	27
6	Monmouth	26
7	Rice	24
7	Hope	24
8	Carleton	21
8	Swarthmore	21
9	Antioch	20
10	Central College (Missouri)	19
11	Wabash	18
12	Wesleyan	17
12	College of Wooster	17
12	Franklin & Marshall	17
13	Mt. Union	16
13	Union	16
13	Juniata	16
14	Haverford	15
14	Hiram	15
14	Reed	15

Institute of Technology and the undergraduate college of the University of Chicago. Outstanding in the list is Monmouth College. From this college better than six of every 100 men graduating have gone on to the doctorate in chemistry (and this is not an estimated figure but one based upon an actual list of men receiving the Ph.D.).

A number of other colleges follow along in second rating, including Mt. Union, Carleton,[3] Wabash, Emory,[3] Beloit,[3] Ottawa (Kansas), Mississippi, Manchester (Indiana), Haverford,[3] Cornell (Iowa), St. Thomas,[3] Wittenberg, and Wooster, in that order.

TABLE 4
Rating of Colleges as Baccalaureate Sources of Chemistry Doctorates

Rating	*Institution*	*Men graduates*	*Chemistry doctorates*	*Earned doctorates, %*
1	Monmouth[a]	420	26	6.2
2	Antioch[a]	414	20	4.8
2	Hiram	315	15	4.8
3	Hope	543	24	4.4
4	Central College (Mo.)	443	19	4.3
4	Oberlin[a]	1165	50	4.3
5	Iowa Wesleyan	244	10	4.1
6	Reed[a]	396	15	3.8
7	Kalamazoo	391	14	3.6
8	Juniata[a]	492	16	3.3
9	St. Olaf[a]	839	27	3.2
9	Swarthmore[a]	667	21	3.2
10	Willamette	386	12	3.1
11	DePauw[a]	1460	44	3.0

[a] On the 1950 approved list of the American Chemical Society.

Ordinarily we associate the motivation in the direction of graduate training to a stimulating atmosphere of research and scientific intercourse. Consequently, we might expect that these 14 institutions were characterized in the thirties and forties by research activity. This is far from the case, as can be seen in the report of J. Sampey.[4] From these we find that only six of the first 14 have published any research papers in the *Journal of the American Chemical Society* in the 23-year period from 1927 to date. Sampey lists in the first reference the 12 most productive colleges as measured by output in this journal; they are Mt. Holyoke, Trinity, Amherst, Barnard, DePauw,[5] Vassar, Swarthmore, Wesleyan, Middlebury, Wellesley, Greensboro, and Union. Interestingly enough, 30 per cent of the research came from five women's colleges on the list—Barnard, Greensboro, Mt. Holyoke, Vassar, and Wellesley. In spite of their activity in chemical research, these 12 colleges were responsible for only 160 chemistry doctorates. The interrelating of the term "research" with the volume of published material tends to the drawing of erroneous conclusions when these refer to the liberal arts college chemistry department. Laboratory work carried out in course may be research, although it may never reach the point where publication is indicated. The research viewpoint may be present and serve as a stimulus to enhance the interest of the student in the course. The informality and small class size of the liberal arts college make this method of teaching easier to carry out, provided the teacher is so minded.

Good laboratories, well ventilated, with pleasant working surroundings, are often given as requirements for any effective program of professional training. The writer is familar with the majority of the chemical facilities of these 14 schools. In the main, they are satisfactory but not extravagant; two at least might be classified as poor. In the laboratories of one of the two, students may be seen carrying on small research projects in odd corners under extremely difficult operating conditions. In spite of the poor facilities, high student morale prevails. Many departments in beautiful buildings produce neither research nor students who are interested in chemistry as a profession.

Somewhat related to the quality of their laboratory buildings are the financial positions of the institutions. Here we find a wide variation, with endowments running from less than a million up to many millions, with a wide spread as well in buildings and grounds. Good salaries generally go with large endowments, poor ones with small endowments.

It is neither high salaries, good laboratories, fine libraries, nor extensive research interests which explain the professional productivity of these schools, although these are in evidence in a number of the group. As the list is reviewed two things stand out. One is the fact that the majority of the schools are located in the midsection of the country (20 of the 27 named in the two lists). But more significant is the fact that these schools have had outstanding personalities as teachers in the chemistry departments—teachers who were interested in the students as individuals and who were alert to the opportunities in the profession of chemistry. The writer of this article is personally acquainted with the personnel of the chemistry departments in many of these schools and can testify to the inspiration and enterprise they transfer to their students. The professor responsible for the record of Monmouth College, W. S. Haldeman, has appropriately been selected for the 1950 Midwest Award of the St. Louis Section of the American Chemical Society.[6]

The particular opportunity of the liberal arts college as an effective place for the undergraduate training of the professional chemist would seem to lie in the close personal relationship possible between teacher and student throughout the entire four-year period. This is more than just the fact that the advanced chemistry classes in the college are generally small in size—the same may be true in the university. The professor who cares will be an important part in the student's adjustment to life in general as well as to chemistry in particular. The professors are "models to be admired

[3] On the approved list of the American Chemical Society.

[4] SAMPEY, J., *J. Higher Education*, **20**, 208 (1949); *Chem. Eng. News*, **28**, 860 (March 13, 1950).

[5] See BLANCHARD, W. M., "Fostering the spirit of inquiry in the college of liberal arts," J. CHEM. EDUC., **12**, 459–61 (Oct., 1935).

[6] *Chem. Eng. News*, **28**, 3436 (Oct. 2, 1950).

and emulated, mustering the energies of their students and crystallizing their ambitions," as Knapp[7] puts it in a recent article. Unfortunately, the numbers of such men are few, and the college is often just another college in the nonproductive sense, seemingly more interested in the morals or pleasure of the student than in the development of his intellectual resources.

[7] KNAPP, R. H., *Wesleyan Alumnus*, page 24 (August, 1950).

DIVISION OF CHEMICAL EDUCATION

OF THE AMERICAN CHEMICAL SOCIETY

118th Meeting, Chicago, Illinois

MEETING OF THE EXECUTIVE COMMITTEE[1]

The following members were present: O. C. Dermer, Oklahoma A. & M. College, *Chairman*; D. G. Nicholson, Pittsburgh; L. L. Quill, Michigan State; James M. Holbert, University of Chattanooga; and P. H. Fall, Hiram College. In addition to the foregoing members of the Committee, the following were in attendance part of the time: M. V. McGill of Howe Academy; E. C. Weaver of Phillips Academy; and T. A. Ashford of University of Chicago.

Mr. McGill, who had requested the privilege of presenting a matter to the Executive Committee, suggested that a committee or a group of interested individuals prepare a series of articles that might be published in the JOURNAL OF CHEMICAL EDUCATION and eventually become the chief material for a textbook in high-school chemistry. Mr. McGill said he is willing to work on the program if the Division will back it. The Chairman said he thought the Committee should take the matter under consideration.

The minutes of the meeting of the Executive Committee that was held in Philadelphia April 11, 1950, and had been sent (along with the agenda for this September 5, 1950, meeting) to the members of the Executive Committee on August 15, 1950, were approved.

The report of the Treasurer, C. E. White, was presented by the Secretary and accepted by the Committee. A copy of the report accompanies this report.

Mr. E. C. Weaver, Chairman of the Committee on High-School Chemistry, reported that his Committee feels that the best service it can render to chemical education at the present time is to undertake the preparation of a small pamphlet on the topic "Motivation in High-School Chemistry," a survey of the means available to popularize the study of high-school chemistry among high-school administrators and students.

He also reported that as of September 1, 1950, the Committee has an unexpended balance of $12.50, and he requested an appropriation of $100 from the Division to finance the current project of the Committee. A motion was passed to receive the report.

Dr. O. M. Smith, Chairman of the Committee on Teaching of College Chemistry, reported that his Committee was responsible for the program of the Workshop in General Chemistry,[2] which was held at Oklahoma A. & M. College, Stillwater, Oklahoma, June 12 through June 23. All (about 55 workers) who attended the Workshop seemed to feel that it was exceedingly worth while and enthusiastically endorsed the proposal that another one be held in 1951. Dr. Smith stated that the total cost of the Workshop was approximately $2,000, $1,500 of which was furnished by Oklahoma A. & M. College. He reported that he has overdrawn by about $100 the $300 that was allocated to his Committee in September, 1946, and asked that another $300 be appropriated to his Committee in order to enable him to liquidate the indebtedness and to continue the work his Committee is undertaking. He also reported that under the auspices of his Committee, Dr. Friedenberg is preparing a manual of suggestions for graduate students who are teaching chemistry. A motion was passed to receive Dr. Smith's report.

Dr. L. L. Quill, who is the Representative of the Division of Chemical Education on the Cooperative Committee on Science Teaching, reported that the Committee sponsored a symposium at the A.A.A.S. program last December and has plans for a program at the meeting of A.A.A.S. to be held in Cleveland in December, 1950. Dr. Quill stated that the Committee is working

[1] Tuesday, September 5, 1950. Room 782, Roosevelt College.

[2] See J. CHEM. EDUC., 27, 619 (1950).

with the U. S. Office of Education in an effort to increase standards of teachers. He also stated that there has been a committee of forty-eight individuals meeting in East Lansing, discussing the question, "How can we evaluate general education?" He spoke of the great loss due to the death of Dr. Shorling of Michigan in May, 1950.

Dr. T. A. Ashford, Chairman of the Committee on Examinations and Tests, reported that 42,000 tests were used last year. He requested that the Division withdraw the request made to the Chemical Education Committee of the A. C. S. in the fall of 1949 for financial support of testing service, because a misunderstanding had arisen due to a discrepancy between the motion passed at the September, 1949, meeting of the Executive Committee of the Division and that made at the business meeting of the Division two days later.

Dr. Ashford also reported that increased prices demanded by the Educational Testing Service seemed to prohibit further negotiations with that organization in the publication and distribution of the chemistry tests. After several possible procedures were discussed, it was agreed that Dr. Ashford discuss the legal phases of the problem with Mr. Emery. Motion passed that Dr. Ashford's report be received.

Due to the lateness of the hour, those present agreed to adjourn and to continue the meeting at the Congress Hotel the following morning at eight o'clock.

At this Wednesday morning (September 6) meeting at the Congress Hotel the following were present: O. C. Dermer, Oklahoma A. & M. College, *Chairman*; J. A. Campbell, Oberlin College; D. G. Nicholson, Pittsburgh; James M. Holbert, University of Chattanooga; N. W. Rakestraw, Scripps Institution; O. M. Smith, Oklahoma A. & M. College; L. L. Quill, Michigan State; E. C. Weaver, Phillips Academy; I. M. Granara, Simmons College; and P. H. Fall, Hiram College.

L. L. Quill of Michigan State College was re-elected to membership on the Board of Publication of the JOURNAL OF CHEMICAL EDUCATION. His term expires September, 1952. William F. Kieffer of Wooster College was elected to membership on this same Board to succeed J. H. Timm of Simmons, whose term expired in September, 1950. Dr. Kieffer's term expires in September, 1953.

Motion passed that the Committee recommends that $17,500 of the funds now accumulated from the JOURNAL OF CHEMICAL EDUCATION be kept in reserve as a backlog for publication of the JOURNAL in a possible "lean" period.

Motion passed that we confirm the action taken by mail that the Division underwrite the publication of an Index of the JOURNAL OF CHEMICAL EDUCATION for the first twenty-five years of its publication.

Motion passed that Frederic B. Dutton of Michigan State be appointed Alternate Councilor to succeed H. H. Sisler of Ohio State whose term expired September, 1950. Dr. Dutton's term expires December 31, 1952.

Motion passed, approving the minor changes made in the Bylaws of the Division, as suggested in a letter from Alden Emery to O. C. Dermer, dated November 7, 1949. In no case was any change suggested or made which in any way altered the intended meaning of the Bylaws of the Division, as published in July, 1949, issue of THIS JOURNAL and passed at the annual business meeting of the Division in Atlantic City in September, 1949, except as noted in the following motion.

Motion passed that the term of office of Councilors be altered to conform with the regulations of the A. C. S. Our Bylaws (III-Sec. 5) stated "The term of officers (which included elected Councilor) shall begin at the close of the meeting at which they are elected." In accordance with above motion this has been changed to read as follows: "The term of officers shall begin at the close of the meeting at which they are elected except for Councilors and Alternate Councilors whose terms shall begin on January 1, following election in the fall, and shall expire on December 31 of the calendar year their respective terms end."

Since, according to our Divisional Bylaws, the Secretary of the Division serves *ex officio*, as a Councilor, Bylaw IV-2-a, which stated "One of the Councilors shall be the Secretary who shall serve *ex officio*," now reads "One of the Councilors shall be the Secretary, who shall serve *ex officio*, except that the Secretary, whose term of office expires at the close of the fall meeting, shall serve as Councilor until the following January 1."

Motion passed that $100 be appropriated to the Committee on High-School Chemistry, E. C. Weaver, *Chairman*, for necessary expenses; that $300 be appropriated to the Committee on Teaching College Chemistry, O. M. Smith, *Chairman*, for necessary expenses; and that up to a maximum of $50 be appropriated to Harrison Hale of University of Arkansas, for clerical and incidental (but not travel) expenses involved in preparation of a history of the Division of Chemical Education. Dr. Hale had agreed to the request made by O. C. Dermer, Chairman of the Division, that he write a history of the Division.

Motion passed that the Treasurer be authorized to pay the following bills:

$7.90 to Edward C. Fuller, for reimbursement for registration fee and divisional luncheon ticket for Dr. Scott Buchanan, a nonchemist and invited guest speaker at the Symposium on Teaching Chemistry to Non-Major Students.

$13.84 to Edward L. Haenisch for reimbursement for telephone calls and telegrams required in connection with the Symposium on Standardized Tests, which he arranged.

$2.90 to Dr. Pauline Beery Mack of Penn State for divisional luncheon ticket, since she was guest speaker.

Motion passed that the Secretary confer with Mr. Emery concerning the possibility of a refund of registration fee for the nonchemists who were invited speakers on symposia programs sponsored by the Division of Chemical Education.

Motion passed that the Division of Chemical Education concentrate its program for the divided spring (1951) meeting of the A. C. S. in Boston (April 1–5), and not attempt to hold a program in Cleveland (April

8-12) also, which is the other of the two cities where the divided meeting is to be held.

For the big 75th Anniversary meeting, which is to be held in New York City, September 2–7, 1951, the following symposia were proposed:

A. Theory of Color, to be chairmaned by Melvin Calvin of University of California, Berkeley.
B. Graduate Instruction, to be chairmaned by Henry Bent of University of Missouri.
C. What to Leave Out of General Chemistry, to be chairmaned by James M. Holbert of University of Chattanooga.
D. Subject Matter versus Student, to be chairmaned by L. L. Quill of Michigan State.
E. Lecture Demonstrations, to be chairmaned by H. N. Alyea of Princeton.
F. College-High School Relationships in Chemistry, to be chairmaned by E. C. Weaver of Phillips Academy.

Meeting adjourned at 9:30 A.M.

PAUL H. FALL, *Secretary.*

REPORT OF TREASURER, SEPTEMBER 1, 1950

Balance on hand 9/1/49		$16,748.75
Removed to Journal Account:		
Bonds	$11,000.00	
Cash	4,559.75	15,559.75
Adjusted balance on hand		$ 1,189.00
Receipts:		
Adjusted balance on hand	$1,189.00	
Dues, 495 members	495.00	
Dues paid in advance	90.00	
Interest on bonds	7.50	
Interest on bank account	27.66	
Total receipts		$ 1,809.16
Expenditures:		
A. C. S. News Service—mailing separates	86.86	
A. C. S.—Dues Receipt Cards	10.50	
Postmaster, College Park—stamps	16.50	
Francis White—typing envelopes	6.75	
U. of Md. Press—envelopes	6.00	
Paul Fall—Office allotment	50.00	
Speakers Dinner at Philadelphia	2.90	
O. Smith—Committee on College Teaching	100.00	
Mack Printing Co.—supplies	33.94	
C. E. White—envelopes and long-distance phone call to Fall	3.96	
Postmaster, College Park—stamps	9.20	
U. of Md. Press—envelopes	6.00	
A. C. S. News Service—mailing separates	56.39	
Total expenditures		389.00
Balance on hand 8/31/50		$ 1,420.16
Assets are distributed as follows:		
Checking account—"Division of Chemical Education," Prince Georges Bank and Trust Co., Hyattsville, Maryland		1,120.16
Three (3) U. S. Savings Bonds, Series G. $100 ea., Nos. C825780g, C825779g, C825778g. Deposited in the safe deposit box of C. E. White at Prince Georges Bank and Trust Co., Hyattsville, Maryland		300.00
Total		$ 1,420.16

Respectfully submitted,
CHARLES E. WHITE, *Treasurer.*

JOURNAL ACCOUNT

Balance on hand 9/1/49:		
Bonds	$11,000.00	
Cash	4,559.75	
Total		$15,559.75
Receipts:		
Chemical Publishing Co.	$ 2,440.27	
Interest on Bonds	325.00	
Total receipts		2,765.27
Balance on hand 8/31/50		$18,325.02
Assets are distributed as follows:		
Savings Account—"Division of Chemical Education," Prince Georges Bank and Trust Co., Hyattsville, Maryland		$ 325.02
Seven (7) U. S. Savings Bonds, Series G. $1000 ea. Nos. M5: 560890g, 560861g, 560859g, 560858g, 560857g, 560856g, 560855g, deposited with Robert V. Mellefont, Treas. *Am. Chem. Soc.*, 1155 Sixteenth St., N. W., Washington 6, D. C.		7,000.00
Four (4) U. S. Savings Bonds, Series G. $1000 ea. Nos. M5: 855178g, 855177g, 855176g, 855081g. (Deposited with R. V. Mellefont, as above.)		4,000.00
Four (4) U. S. Savings Bonds, Series G. $1000 ea. Nos. M7: 280064g, 280066g, 280067g, 279936g. (Deposited with R. V. Mellefont, as above.)		4,000.00
Three (3) U. S. Savings Bonds, Series G. $1000 ea. Nos. M7: 438010g, 437661g, 438009g. (Deposited with R. V. Mellefont, as above.)		3,000.00
Total		$18,325.02

MINUTES OF THE BUSINESS MEETING[3]

The meeting was called to order at 11:30 A.M. by the Chairman, O. C. Dermer. In the absence of the Secretary, Paul H. Fall, Dr. N. W. Rakestraw served in that capacity.

The minutes of the previous meeting were approved as published. The report of the meeting of the Executive Committee, including the Treasurer's report, were read and approved.

The Chairman announced the following Committee appointments:

Committee on Affiliation of the PSACT[4]: E. C. Weaver, *Chairman;* H. E. Redeker; J. B. Entrikin; L. L. Quill.

Committee on Proposed Code for Engaging Graduate Assistants in Chemistry: R. K. Summerbell, *Chairman;* A. W. Laubengayer; John E. Willard; Robbin C. Anderson; James F. Corwin.

The Nominating Committee, comprised of L. L. Quill, *Chairman*, John C. Bailar, and E. L. Haenisch, presented the following nominations, which were duly elected.

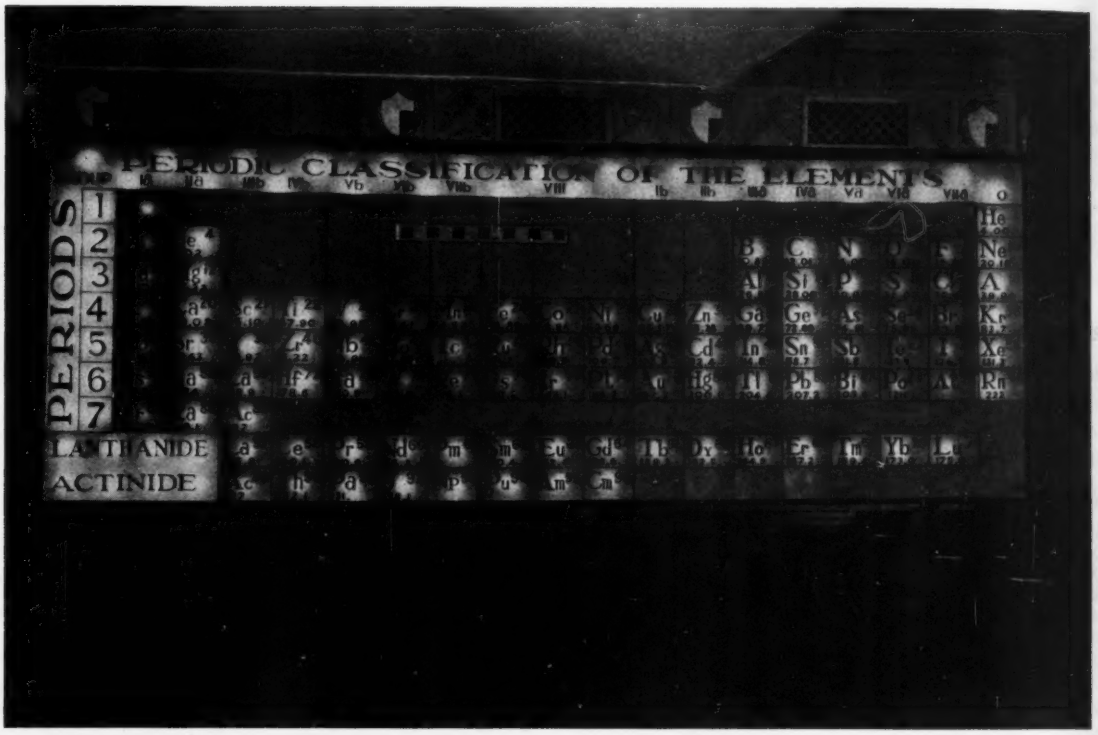
Chairman	J. A. Campbell, Oberlin College
Chairman-elect	H. N. Alyea, Princeton University
Treasurer	F. D. Martin, Purdue University
Elected Councilor	J. Kleinberg, University of Kansas

NORRIS RAKESTRAW, *Acting Secretary for Business Meeting*
PAUL H. FALL, *Secretary*

[3] Thursday, September 7, 1950. Sinha Memorial Hall, Roosevelt College.
[4] Pacific Southwest Association of Chemistry Teachers.

A LECTURE ROOM PERIODIC TABLE[1]

FREDERIC B. DUTTON
Michigan State College, East Lansing, Michigan



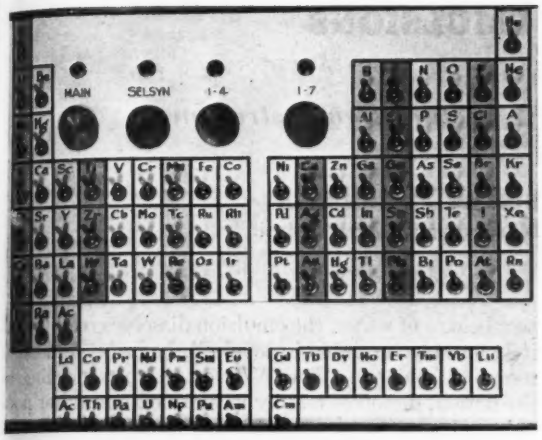
To satisfy a desire for a lecture room periodic table which would be visible to all students in the room, which would permit focusing attention on a desired item, and yet would not be so complex as to be distracting, there has been erected in the lecture room of the Kedzie Chemical Laboratory at Michigan State College the periodic table shown above.

The data for the table are painted on ground glass mounted in front of a network of metal boxes housing $7\frac{1}{2}$-watt light bulbs. The titles, period numbers, and group numbers are illuminated when a master switch is closed. By closing individual switches on a control panel at the lecture table the symbols for each element may be illuminated.

The electronic configuration for any element under discussion can be indicated in the seven squares shown in the open space above Cr, Mn, Fe, and Co in the table. This is done by illuminating the openings from the rear and passing a tape carrying the desired numbers next to the glass. The tape is passed from one spool to another by rotating the drive spool with a selsyn motor which is controlled by a similar motor on the control panel. The idler spool is operated by a counter weight. Motor driven tapes are behind the four openings to the right. The three openings at the left carry the fixed numbers 2, 8, 18 painted on the glass and are used only when elements in the fifth period and higher are being discussed, *i.e.*, when more than four numbers are required to express the electronic configuration, in which case the first three electronic levels are full.

The table is supported from two-by-fours bolted to the wall and carrying a $\frac{3}{4}$-inch plywood back. A heavy 16-gage galvanized iron channel forms the outer periphery. It is $5\frac{1}{2}$ inches deep with the front side 1 inch wide at the sides and bottom and $1\frac{1}{2}$ inches wide at the top. This permits the glass sections to be raised and swung out at the bottom for removal. The plywood back was covered with sheet aluminum and the net-

[1] Presented at the 118th meeting of the American Chemical Society in Chicago, September, 1950.

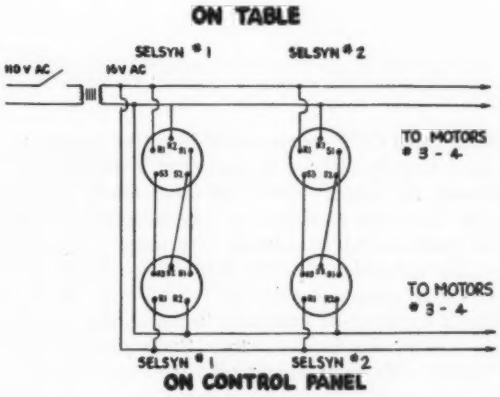


Lecture Table Control Board

work of boxes, which are $6^1/_2 \times 9^1/_2$ inches each, constructed of 22 gage galvanized iron strips running vertically. The horizontal dividers were of the same material and were fastened in place with sheet metal screws after a hole was punched in the center of the rear edge for a rubber grommet, through which the electrical connections were made. The depth of the boxes was $5^9/_{16}$ inches and was calculated so that there would be just room for the $^3/_{16}$-inch glass to rest against the boxes at the back side and be retained in place by the channel frame in front, thus holding the glass in place. The glass was cut in strips, each strip carrying two groups of elements with one narrower strip at the left for the period numbers.

The $7^1/_2$-watt light bulbs were mounted in standard cleat receptacles in the center of the boxes and similarly spaced for the titles. The bulbs were dipped in Christmas tree lamp lacquer that had been thinned and tinted by mixing the various colors available. Each group was assigned a different color.[2] A thin layer of glass wool was placed in each box over the light bulbs to increase the diffusion of the light and minimize bright spots. The lettering was painted in reverse on the smooth side of the glass and the frosted side was exposed to avoid reflections. The space over the 7 squares for the electronic configuration was lacquered to make it transparent.

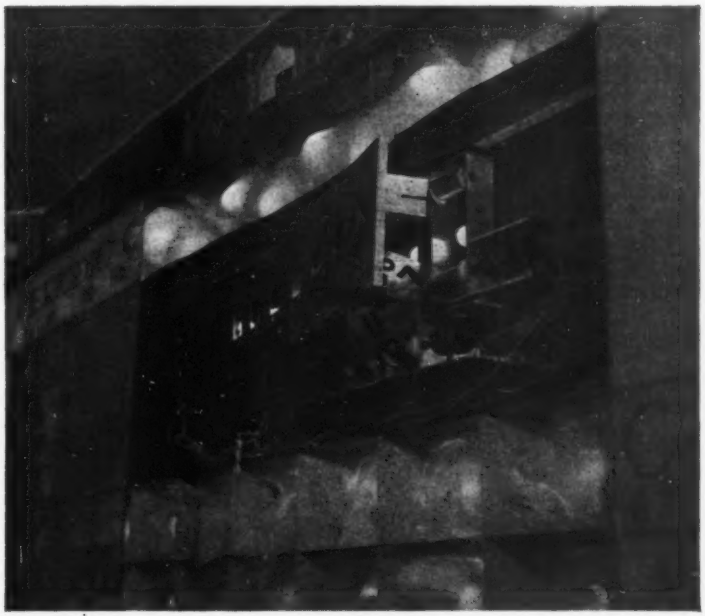
All lamp sockets were wired with one side in common. All socket terminals were brought to a connector strip at the top of each group, from which a multiwire cable led to the control panel on the lecture table. The selsyn motors were driven through a transformer. They are rated at 110 volts, 400 cycles but operate satisfactorily at 16 volts on 60 cycles. They are the war surplus Model 2JD1J1. The selsyn motors are manufactured by General Electric and others.



Wiring Diagram

ACKNOWLEDGMENT

Grateful acknowledgment for assistance is due to many members of the staff and particularly to Drs. Byerrum, McCarty, Pitha, Stone, and Schuetz, who took many evenings away from their families and their research to help build the assembly.



Motors and Tape Mechanism

[2] Kodachrome slides of the table and control panel are available from the writer on loan.

OIL AND WATER EMULSIONS

A Lecture Demonstration

SISTER M. IGNATIA
Marygrove College, Detroit

THE symbol O/W means an oil-in-water emulsion, one in which tiny droplets of oil, colloidal in dimensions, are dispersed in water. Since an emulsion has approximately the same solubility as its continuous phase, an O/W emulsion can be diluted with more water (a commercially valuable property) but is insoluble in more oil or in other nonpolar solvents. Similarly, W/O symbolizes a water-in-oil emulsion in which the continuous phase is nonpolar in character. Such an emulsion can be diluted with more oil but not with more water.

To form stable emulsions of these two types, surface-active agents are needed, compounds whose molecules are not only complex, but in which one part of each molecule is hydrophilic or water-loving, and another part is lipophilic, or oil-loving. Sometimes surface-active agents are more effective when used in pairs. This is true if one is strongly attracted to water, the other to oil, and the agents to each other. Among the surface-active agents commercially available are the Span and Tween products of the Atlas Powder Company. The Spans tend to be oil-soluble and the Tweens water-soluble. Properly selected, a Span and a Tween together make an effective emulsifying agent. According to the booklet, "Atlas Surface Active Agents," the "Span type materials are essentially partial esters of the common fatty acids (lauric, palmitic, stearic, and oleic) and hexitol anhydrides (hexitans and hexides), derived from sorbitol. The Tween type materials are derived from the Span products by adding polyoxyethylene chains to the non-esterified hydroxyls. Both types are actually complex mixtures of several compounds."

To illustrate the formation and the solubility of O/W and W/O emulsions, I have worked out Dr. George E. F. Brewer's idea for a simple demonstration experiment in general chemistry. We prepare an O/W emulsion by putting 100 ml. of distilled water into the semimicro head of a Waring blendor, starting the blendor, and adding 6.6 ml. of Tween 81, 7 ml. of Span 85, and, slowly, 100 ml. of level oil. Mineral oil can be used instead of level oil, but the latter is less expensive and its flash point (120°F.) is not likely to be reached.

In preparing the W/O emulsion, 100 ml. of level oil are put into the blendor, the blendor started, and 7 ml. of Span 85, 6.6 ml. of Tween 81, and, slowly, 100 ml. of distilled water are added. Both emulsions are poured into beakers and their volume, color, texture, and viscosity compared.

When a few milliliters of the O/W emulsion are added to a beaker of water, the emulsion dissolves readily, but it is notably insoluble in level oil, benzene, and other nonpolar solvents. The W/O emulsion, according to prediction, dissolves readily in more oil and other nonpolar solvents, but is insoluble in water.

Dreft acts as an emulsifying agent for an O/W emulsion but is not satisfactory for the W/O type. One-fifth of a gram of Dreft is sufficient to hold 100 ml. of oil in 100 ml. of water. Other pairs of Spans and Tweens, for example, 20 and 20 or 60 and 60, may be substituted for Span 85 and Tween 81, but the last pair seems to give the most stable emulsions of the W/O type and is quite satisfactory for demonstration of O/W emulsions.

If the students need to be convinced that emulsions formed without the aid of emulsifying agents are unstable, 50 ml. of level oil may be added slowly to 50 ml. of water in the blendor, or vice versa. The emulsion formed is so unstable that it separates into layers even before it can be tested as to type.

If time permits, the emulsions prepared during the demonstration may be reversed in phase. If water is put into the blendor and part of the W/O emulsion added while the blendor is running, the emulsion is readily reversed to a stable O/W emulsion. O/W emulsions formed with the Spans and Tweens reverse, but the resulting W/O colloidal suspensions tend to be unstable. O/W emulsions formed with Dreft do not reverse in phase to W/O, probably because Dreft is not a satisfactory emulsifying agent for the latter type. If the blendor containing oil and an O/W emulsion formed with Dreft is stopped and the oil layer allowed to come to the top before the motor is started again, a stable and stiffer O/W emulsion is formed.

Students are frequently amazed to learn that a stable emulsion may be formed in which the continuous phase is considerably smaller in volume than the dispersed phase. If, for example, 20 ml. of level oil are put into the blendor, the blendor started, and 4 ml. each of Span 85 and Tween 81, and 80 ml. of water added, the last very, very slowly, a stiff and stable W/O emulsion is formed, in spite of the fact that the dispersed phase has four times the volume of the continuous phase. Similarly, a stable O/W emulsion in which the volume ratio of dispersed to continuous phase is four to one can be formed by putting 20 ml. of water and 0.5 g. of Dreft into the blendor and adding 80 ml. of oil very, very slowly while the blendor is running.

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

HORMONES—PLANT GROWTH REGULATORS[1]

P. W. ZIMMERMAN
Boyce Thompson Institute for Plant Research, Inc., Yonkers 3, New York

THE subject of this paper involves one of the newer horizons in science—chemicals which regulate the growth of plants. As a concept lying along the frontiers of science, plant hormones appeared on the horizon with the publication of a book in 1880 by Charles Darwin called "The Power of Movement in Plants." Very little progress was made during the first fifty years, but startling advances occurred during the last twenty years. Since the discovery by Boysen-Jensen that phototropic curvatures in plants were induced by an influence of a chemical nature, rapid progress has been made.

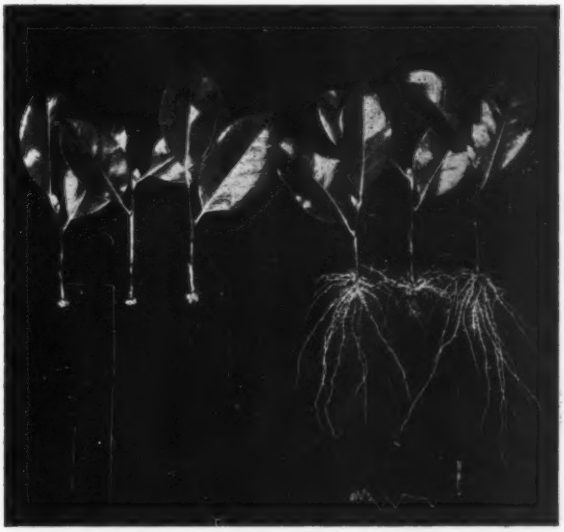
The term "hormone" for plants is not well defined. From a popular point of view the word means any influence of a chemical nature which regulates the growth of plants. The term "growth substance" or "auxin" is often used to mean the same as "hormone."

Interest in the subject of plant hormones from the standpoint of fundamental science is still uppermost in the minds of many workers. It is increasingly evident, however, that an attack on practical problems in the field of plant hormones brings about results that contribute to both science and practice. Laboratory curiosities pointed the way to horticultural applications, and we now use plant hormones to propagate plants, prevent preharvest drop of apples, inhibit buds to prevent potatoes from sprouting, increase fruit set and induce seedless tomatoes, regulate flowering of pineapples, kill broad-leaved species without killing grass, and defoliate plants without killing the stems.

Groups of chemicals best known as growth regulators are as follows: indole compounds involving β-indoleacetic and β-indolebutyric acids; naphthalene compounds involving α-naphthaleneacetic acid and its derivatives; β-naphthoxyacetic acid and higher homologues; substituted phenoxyaliphatic acids, esters, and salts, especially 2-chlorophenoxyacetic acid, 4-chlorophenoxyacetic acid, 2,4-dichlorophenoxyacetic acid, 2,4,5-trichlorophenoxyacetic acid, and their higher homologues; and substituted benzoic acids involving especially 2,3,6-trichlorobenzoic acid, 2,3,5-triiodobenzoic acid, and 2,5-dichlorobenzoic acid. To these should be added unsaturated hydrocarbon gases, especially ethylene.

There are many natural hormones which have not been isolated and identified but, nevertheless, are known to exist. Indoleacetic acid and ethylene, mentioned above, are made by plants and have been identified by chemical methods.

Many workers are trying to find how hormones work and how they are synthesized by plants. Physiological activity is found with simple chemical structures like ethylene as well as the more complex structures like



Root Inducing Power of Indolebutyric Acid when Applied to *Camellia japonica*

Left—Not treated.
Right—Treated with indolebutyric acid in talcum powder.

[1] Based on a paper presented at the Twelfth Summer Conference of the New England Association of Chemistry Teachers, University of Connecticut, Storrs, Connecticut, August, 23, 1950.

View of Waterway in Louisiana Infested with Water Hyacinth

α-naphthaleneacetic acid and substituted phenoxyaliphatic acids. New physiologically active compounds are being discovered almost daily, and it is likely that the most effective ones are yet to come.

Extensive uses of plant hormone-like substances became evident during the past year when the sale and use of 2,4-D (2,4-dichlorophenoxyacetic acid) practically equaled that of the insecticide DDT which has been leading the list of all organic agricultural chemicals. This new selective herbicide is now being used to control weeds in farm crops, yards, golf courses, and marine growth in swamps and waterways of the south. New uses are being found for 2,4-D, and its sales may have exceeded that of DDT in 1950. Weed killing is by far the most important practical use of hormone-like chemicals. This alone has a potential annual market of more than 100,000,000 pounds. The second in importance is the use of naphthaleneacetic acid and its derivatives to prevent preharvest drop of apples and inhibit bud growth of potatoes.

The Appearance of a Stream after Treatment with 2,4-D.

There are also other achievements with plant hormones, and some of these illustrate the varied lines of attack from the scientific and practical point of view. In Hawaii several kinds of plant hormones are being used to force pineapples into flower or to increase fruit size. Still other hormones prevent premature flowering, and finally vegetative propagation of pineapple is facilitated with plant hormones. Induction of roots on cuttings was one of the early practical uses found for plant hormones. The same chemicals which induce roots to form may also inhibit growth of other parts of the plant.

Let us try to look beyond the present horizons for plant hormones and predict some of the things to come. We shall have hormone-like selective weed killers for every conceivable use—the corn fields, the wheat fields, the orchards, the gardens, the forests, the waterways, the swamp lands, the hedge rows, and the yards. Any hormone which has specificity in its effects is likely to find special uses. Perhaps the forest can be sprayed to kill all but pines or other desired species. With a little stretch of the imagination we can picture a lawn without crabgrass, a bayou in Louisiana without water hyacinth and alligator weeds, pastures without thistles, onion fields not weeded by hand, and helicopters available for spraying at 25 cents an acre.

Bud-inducing chemicals are seriously needed. They would facilitate propagation of plant parts where buds have not appeared or where they have been lost. Day lilies, Gloriosa lilies, and dahlias are often lost because the storage organ does not have a natural bud. Internodes which do not normally produce adventitious buds could be used for propagation purposes. A shoot-inducing substance should cause new shoots to arise where desired on the plant to make possible propagation of budless parts or improve the shape and appearance of intact plants. There is no end to the conceivable uses of bud- or shoot-inducing substances.

Flower-forming substances which are thought to exist in nature may be isolated, identified, and used as a common tool. If this becomes a reality it should be possible to force long-day types to flower during short days or short-day types to flower during the long days. In short, it should be possible to induce flowering of plants at will and to force flowers to grow at unusual places. We can imagine plants with flowers on internodes, on leaves, and even on roots.

At the present time there appears to be considerable variation in the time of ripening of fruit. Under the influence of chemicals the time of ripening should fit into our needs. During the past season it has been shown that apples treated with certain hormone-like chemicals ripened prematurely. In a lesser degree this has been noticed for tomatoes. Tomato flavors are not affected under the influence of the chemical. Apples, however, change flavor and consistency, but the very fact that modifications in time of ripening have been demonstrated offers encouragement for practical methods applicable to all or many fruits.

Since it has been demonstrated that fruit buds can be

delayed through treatment with growth substances, one is led to the assumption that fruiting of tropical species and flowering of plants in general can be staggered to extend throughout the entire season. Mangoes, for example, flower and ripen fruit at very definite periods of the year. During the rest of the year they are not available as food. Since this is an important tropical food it would be desirable to extend it throughout the season. This should become a reality by the proper hormone applications to growing buds. The idea is particularly applicable to tropical plants, because the temperature and other growing conditions would not limit the time of fruiting. We should, however, be also able to stagger flowering or spring shrubs in the north so that we could enjoy them over longer periods.

Without making further predictions, it appears that the field of plant hormones presents a challenge for scientists with varied attacks. As far as fundamental research is concerned, it might be said that the work is just beginning. We have found only a few natural hormones, and we still know little about how hormones work. From the standpoint of horticultural applications, progress has been more rapid than in fundamental research, and the results are so spectacular that interest in the subject is now sweeping the world. Research holds much for the future and more important applications are sure to be made.

There is much competition in the fields covered by this paper. Perhaps that is why progress has been rapid. Research in some phases of the subject can be done by amateurs and practical horticulturists as well as by trained scientists. It is well to recognize the fact that several techniques have played a part in solving difficult problems. We take new courage when we remember that this is the age of research. We must follow where research leads. Thousands of researchers are looking into the unknown or fields that have no other limits than man's imagination. Research is not confined to the laboratory. Its origin is in the individual. All of us are researchers no matter what our jobs, and we must surpass what has already been accomplished to keep ahead. We must search for new and better methods, for even that which we now do well must be done better tomorrow.

To the Editor:

Any mnemonic device that will assist the beginning student to correlate information on the gas laws is to be welcomed, and I noted the chart proposed by Calvin P. Midgley with interest. The spelling of the name of Gay-Lussac should be corrected. The omission of Graham's law was a surprise. Perhaps no appropriate key word suggested itself. Why not the "A, B, C, D, and G's" of the Gas Laws? The key word for Graham might be "Going." This would connote the motion of gaseous molecules.

It is more probable, however, that the author is following the fashion of the day. In certain of the textbooks in general chemistry published in the last few years the name of Graham is not mentioned in the discussion of the diffusion of gases, and the formulation of the law is also often omitted (see 1950 editions of the textbooks by L. Pauling and H. I. Schlesinger). What is the explanation for this omission? Graham's law is not essential in developing the topic of gases that is to culminate in the concept of gram-molecular volume. But it seems to me that without at least some mention of it much of value is lost in the presentation of the kinetic-molecular theory of gases. With what experimental evidence is the equation K.E. $= {}^1/_2\ mv^2$ correlated? There is no doubt that the law is difficult to teach; there is no question that students often fail to solve even simple problems illustrating the law; but the fact that the rates of diffusion of gases vary inversely as the square roots of their densities should be brought to the attention of students because of its application in the problems of the pollution of the air in large industrial and commercial cities today. Perhaps one of the authors who has omitted Graham's law from his textbook will discuss his reasons for doing so in the JOURNAL OF CHEMICAL EDUCATION.

CLARA DEMILT
NEWCOMB COLLEGE, TULANE UNIVERSITY
NEW ORLEANS, LOUISIANA

Recent Books

A PRACTICAL SURVEY OF CHEMISTRY

Walter S. Dyer, late of the University of Arkansas, and *Manfred E. Mueller,* City College of San Francisco. Revised edition. Henry Holt and Co., New York, 1950. x + 564 pp. 178 figs. 13 tables. 14.5 × 21 cm. $3.60.

THIS revision of Dr. Dyer's book which initially appeared in 1941 is greatly improved in appearance: use of smaller size type with more spacing between lines adds to the readability, and use of paper of better quality with semigloss finish results in much better reproduction of the diagrams and illustrations, many of which are new and well-chosen. It is a pity that the several topics considered in each chapter were not numbered to facilitate reference and assignment.

There are few major alterations, although sections of several chapters have been rearranged and interchanged with resulting improvement in clarity and in the order of presentation of related subjects. Thus, a new chapter combines the topics electromotive series, electrolysis, cells, plating, oxidation-reduction, photography, and the light metals. Also the section dealing with organic chemistry has been largely rewritten. Several new topics have been introduced, including silicones, exchange resins, antibiotics, antimalarials, and the Fischer-Tropsch reaction; and two new chapters on nuclear chemistry have been added. The style remains readable and the approach mature.

Designed for courses in general education rather than for training the professional chemist, this book represents one of the best efforts to compromise between a frankly superficial survey of chemistry and a detailed consideration, such as is desirable for the potential chemist. Although it is about chemistry, it adheres to fundamentals with a minimum of arithmetic, and holds the reader's interest by emphasizing applications of principles and theory to every day phenomena. The book deserves consideration by anyone teaching chemistry as a part of a liberal arts program.

ROBERT M. SHERMAN

BROWN UNIVERSITY
PROVIDENCE, RHODE ISLAND

THE BIOCHEMISTRY OF THE B VITAMINS

Roger J. Williams, Robert E. Eakin, Ernest Beerstecher, Jr., and *William Shive,* University of Texas, Austin, Texas. Reinhold Publishing Corporation, New York, 1950. x + 741 pp. 29 figs. 50 tables. 15 × 24 cm. $10.

EACH of the four authors has written a separate section which could be a book in its own right, although each includes interrelations between the subdivisions of the subject. The avowed purpose of the book is to provide a critical survey of biochemical research on the B vitamins. It covers, especially, literature of the years 1945 to 1948 inclusive; but some papers significant in the historical development of knowledge about B vitamins are referred to briefly, and some papers appearing in 1949 or even 1950 are reviewed.

The book uses so extensively data on intermediary metabolism as studied by the use of microorganisms that it is especially helpful to biochemists who have concentrated on mammalian metabolism. The role of these vitamins in higher organisms, including man, however, is not neglected.

The first section by Williams has five chapters dealing, respectively, with the characterization, distribution, extraction (combined forms), assay, and biogenesis of B vitamins. His unique definition of B vitamins, coming as it does from so authoritative a source, will interest all biochemists.

The second section by Eakin has three chapters dealing with the catalytic functions of the B group. This might be regarded as the core of the book since all sections deal more or less with the metabolic functioning of these vitamins.

The third section by Beerstecher has six chapters on the role of the B vitamins in animal and plant organisms. Among the subjects dealt with are quantitative needs for each of the several vitamins as required by many different organisms and the metabolism of the vitamins. The chapter on physiological, pharmacological, and toxicological effects was of especial interest to this reviewer.

The fourth and final section by Shive is a long one of twelve chapters. It deals with the specificity of each vitamin, *i. e.,* the differences and analogies between the effects of the vitamin and those of related compounds. Also for each vitamin there is an admirable review of interference studies, including all aspects of enzyme blocking by compounds related to the vitamins. There is a separate chapter for each of eight of the B vitamins. One of these, the chapter on thiamine, and the last chapter on "nutritional factors of doubtful status" are contributed by writers not listed on the title page, Thomas J. Bardos, A. D. Barton, and Lorene L. Rogers.

This book will be found to be indispensable to researchers and other students of vitamins and to nutritionists.

PHILIP H. MITCHELL

BROWN UNIVERSITY
PROVIDENCE, RHODE ISLAND

OXIDATION-REDUCTION POTENTIALS IN BACTERIOLOGY AND BIOCHEMISTRY

L. F. Hewitt, Director, Serum Research Institute, Medical Research Council, Carshalton, Surrey, England. Sixth edition. Williams & Wilkins Company, Baltimore, 1950. viii + 215 pp. 44 figs. 31 tables. 18 × 25 cm. $4.50.

THIS latest edition is considerably larger than its most recent predecessor, which appeared in 1948, twelve years after publication of the fourth edition. The first two chapters on the theory of oxidation-reduction reactions and on the methods for measuring oxidation-reduction potentials have undergone very little change. The third chapter is new; it gives a brief account (8 pages) of different methods for measurement of pH. In the fourth chapter are treated the different oxidation-reduction systems which are of biological interest; here much of the recent work has been included. The fifth chapter (12 pages) is a newly added discussion of metabolic cycles and chain reactions with a consideration of the significance of oxidation-reductions, free energy changes, phosphate bond energies and energy storage mechanisms. The bacteriological applications are given in the sixth chapter which, in spite of relatively few additions since 1936, is still the most important of the book. In the new seventh chapter (17 pages) an account of chemotherapy and antibiotics is given, and an attempt is made to show that even in this field potential measurements may be of value. It is followed by two brief chapters, one on the polarograph (11 pages) and one giving general conclusions (7 pages). A bibliography of well over a thousand titles as well as author and subject indexes add to the usefulness of the book.

Any reader familiar with the fourth edition of this book will probably share the reviewer's disappointment in the present monograph, because it shows that in the intervening years very little has been added in this field to an understanding of the fundamentals. For instance, the following statement (p. 105) has remained completely unchanged: "Definite electrode potentials are

established in cultures, but we are not fully aware what substances are responsible for these potentials in many cases, nor what reversible oxidation-reduction systems are actually being measured."

The material added in the fifth and seventh chapters actually contributes nothing new to the main subject besides emphasizing the fact that in all biological systems oxidation-reduction potentials do play a role. This broadening of scope, as stated in the preface, "has necessitated the epitomising of large subjects in small compass." Perhaps the author has gone too far in this respect, or should think of another title for the book. For instance, discussions of the Warburg manometer (under Systems of Biological Interest), of microbiological assay (under Bacteriological Applications), and of Fischer water determination (under The Polarograph) seem out of place in this book. Inclusion of this and other material made it necessary to save space by a mere listing of some of the more pertinent contributions in the major topics. For instance, catalysis, the theoretical or practical importance of which cannot be denied, is given hardly more than a page, and the whole topic of reaction kinetics is completely ignored.

Nevertheless, the book, as its predecessors, will be of great help to the bacteriologist and the student of chemistry who wishes to get a broad viewpoint of oxidation-reduction phenomena and their proved or possible importance in all kinds of biological phenomena.

OTTO H. MÜLLER

STATE UNIVERSITY OF NEW YORK
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• PRINCIPLES OF IONIC ORGANIC REACTIONS

Elliot R. Alexander, Assistant Professor of Chemistry, University of Illinois, Urbana, Illinois. John Wiley & Sons, Inc., New York, 1950. viii + 318 pp. 16 × 24 cm. $5.50.

". . .IT IS. . .possible to break down a very large number of organic reactions into. . .a few basic transformations. . . ." The sequence by which these changes take place "is commonly called a *reaction mechanism.*" With this introduction, Alexander takes us through 295 pages of explanations, illustrations, and arguments, and at the same time brings us up to date on the latest theories and experimental facts concerning organic reactions. His approach, as the title tells us, is ionic in nature. In some cases, as in the Wurtz reaction, which is usually explained on the basis of the formation of free radicals, he shows that an ionic mechanism is also plausible. A complete discussion of free radicals is apparently beyond the scope of this book. Some may feel that this omission is a drawback to an otherwise excellent book. Many questions are left still unanswered; yet so much is covered that the student, the research chemist, and the industrial chemist will all find something useful and instructive. After reading this book, low yields and side reactions are more easily understood, and the reader feels that he can visualize and "understand" these reactions to a greater extent. He is also in a better position to predict the probable outcome of new experiments. Let us appreciate, however, that there is no substitute for actual laboratory experimentation.

The book is well thought out; it is also well written. The introduction is good; the second chapter on electrons is better. Here a practical treatment of resonance is given with the good feature that although the usefulness of this phenomenon is shown, no attempt is made to explain what resonance really is. Thus we are not disconcerted by reading many ambiguous explanations. Unfortunately, some of the terms used are at variance with other texts since there is no standardized terminology in this field. Alexander realizes that nomenclature is a stumbling block; in a few cases he has not helped much.

The chapter on Carbonium Ions gives a good picture of the possible steps and paths by which reactions take place. As the theme is developed many common name reactions are analyzed, as are many of the lesser known reactions. Rearrangements are especially well treated. Perhaps a little more evidence for the existence of carbonium ions would be in order. In the chapters on Displacement Reactions and Elimination Reactions some new information is gleaned, and S_N reactions are clearly explained. The organic student may have to be helped quite a bit with the physical chemistry. The background given by the preceding chapters makes the treatment of carbanions more easily understood. Discussions following on noncarbanion addition, especially carbonyl group addition with ammonia derivatives, are among the best in the book. Here students should be able to follow this material with no difficulty. Esterification, hydrolysis, substitution on the benzene ring, and diazonium reactions are also treated in subsequent chapters. The last chapter treats some of the newest work in organic reactions, and mention is made of the work of almost every research chemist presently engaged in the field. Ample references throughout the book encourage further reading.

Unfortunately, space probably did not permit a discussion of the apparent variance of modern theory with classical work. Many references state that secondary amines are almost specific catalysts for the Knovenogel reaction. "It is interesting that, of the number of catalysts and conditions investigated, acetamide in glacial acetic acid proved to be the best." Wherein does the explanation lie? Surely simple dehydration is not the answer.

For a course which has as its prime objective the interpretation of organic reactions and as an introduction to current research the book can be highly recommended.

UNIVERSITY OF SAN FRANCISCO
SAN FRANCISCO, CALIFORNIA
ARTHUR FURST

UNIVERSITY OF SANTA CLARA
SANTA CLARA, CALIFORNIA
JOSEPH F. DECK

• ENCYCLOPEDIA OF CHEMICAL TECHNOLOGY VOLUME V: DI- TO EXPLOSIONS

Edited by *Raymond E. Kirk* and *Donald F. Othmer,* Professors and Heads, Departments of Chemistry and Chemical Engineering, respectively, at the Polytechnic Institute of Brooklyn. Assistant Editors, *Janet D. Scott* and *Anthony Standen.* The Interscience Encyclopedia, Inc., New York, 1950. xvi + 992 pp. 19 × 27 cm. $20.

WHAT was to be the midpoint of E.C.T. has now become just another volume, though equally important and welcome. The total of 1947 pages devoted to the letter "C" was one factor that caused the expansion of E.C.T. from the originally-planned ten volumes to a twelve-volume work. Certainly no one can object to this increased coverage by the high-quality articles appearing in E.C.T.

A successful encyclopedia must have both short and long entries; both must be authoritative. Hence due credit should be given to all authors and all sections. However, the reviewer would like to mention some of the longer sections and their authors, so that their eminence in their fields may be noted.

Several chemical engineering unit operations are included. The chapter on diffusion separation methods (58 pages) is by Manson Benedict, of Hydrocarbon Research, Inc., who received the 1947 W. H. Walker Award of the American Institute of Chemical Engineers, and whose book in this field is well known. Evaporation (22 pages) was written by W. L. Badger, 1940 recipient of the W. H. Walker Award, whose books are extremely well known to chemical engineers. Distillation (31 pages) was prepared by E. G. Scheibel, of Hoffmann-LaRoche, Inc., who received the 1949 Junior Award of the A.I.Ch.E. Drying (34 pages) was written by W. R. Marshall, Jr., associate professor of chemical engineering at the University of Wisconsin, whose papers in the field of drying and book on applications of differential equations are well known. The broad field of Chemical Economics (20 pages) was discussed by Chaplin Tyler of the develop-

ment department of the du Pont Company, whose book on Chemical Engineering Economics has been used widely since 1926. Dimensional Analysis (9 pages), a subject of importance to chemical engineers, was prepared by D. Q. Kern, director of the process engineering division, Patterson Foundry and Machine Company.

Electroplating (36 pages) was discussed by C. L. Faust, Battelle Memorial Institute, who is the new president of the Electrochemical Society. Electrostatic Precipitation (17 pages) was prepared by L. M. Roberts, assistant technical director of the Research Corporation. Organic Electrosynthesis (6 pages) is the special field of Sherlock Swann, Jr., of the University of Illinois, whose papers in this field are well known. Electrochemistry (55 pages), as the physical chemist prefers it, was the speciality of G. C. Akerlof, formerly of Yale University and now of Mellon Institute. Electronics (51 pages) is the everyday field of G. A. Morton and M. L. Schultz of the R.C.A. Laboratories. Dielectrics (25 pages) was prepared by E. B. Baker and W. C. Goggin of the plastics division of the Dow Chemical Company.

Of interest to every organic chemist will be the chapter on Esterification (42 pages) by C. E. Leyes, head of the development and research department of the plastics division of the Celanese Corporation of America. Organic esters (27 pages) are discussed by T. E. Jordan, now a research supervisor for the Barrett Division of Allied Chemical and Dye Corporation. A group discussion of Ethers (18 pages) finds Mary Magill, of Chemical Abstracts, cooperating with J. G. Park and C. M. Beamer of the Enjay Company, Inc. The preparation of that basic organic raw material, Ethylene (18 pages), is discussed by John Happel, previously of Socony-Vacuum Oil Company and now professor of chemical engineering at New York University. Ethylene Amines, by John Conway, of Carbide and Carbon Chemicals Division, and Ethylene Oxide by R. S. Aries and H. Schneider, consultants, and K. Gerzon of Eli Lilly and Co., are products of the ethylene group. The lengthy section on Dyes, intermediates, application, and evaluation (119 pages) is the combined effort of eleven workers mainly from General Aniline and Film Corporation's General Aniline Works Division and General Dyestuff Corporation. One exception is Calco Chemical Division's August Merz, a man whose long experience in the dyestuff industry eminently qualifies him to write on the history of the industry. A name not to be overlooked by organic chemists is Oliver Kamm, of Parke, Davis, and Company, who prepared the section on Epinephrine.

Biochemists will be particularly interested in the sections on Enzymes (28 pages) by A. K. Balls, of the U. S. Bureau of Agricultural and Industrial Chemistry, and on Eggs (13 pages) by A. L. Romanoff, of Cornell University.

The Theory of Explosions and Gaseous Explosions are discussed by G. B. Kistiakowsky, professor of physical chemistry at Harvard University and H. F. Coward, one time of the U. S. Bureau of Mines and now of the British Safety in Mines Research Board.

The reviewer again wishes to stress the importance of all sections and all authors and express the regret that space limitations prevent mention of all. Certainly all have contributed handsomely to making a worth-while volume and set of E.C.T.

KENNETH A. KOBE

UNIVERSITY OF TEXAS
AUSTIN, TEXAS

● WINES OF THE WORLD

Peter Valaer, **Senior Scientist of the Bureau of Internal Revenue. Abelard Press, Inc., New York, 1950. 576 pp. 25 tables. 14.5 × 22.5 cm. $6.50.**

INCLUSION of this book in the general field of chemical interest results from the fact that the manufacture of fermented beverages was one of the earliest chemical industries. Further, the book is an interesting addition to the nontechnical literature of food materials. The author discusses the various wine-types of the world and the "geography" of their production in fascinating fashion. The chemistry in three "technical" chapters is rudimentary, but that on Methods of Analysis of Wine goes into greater detail.

● ATOMIC PHYSICS

Wolfgang Finkelnburg. **Translated from the revised German edition in cooperation with the author by George E. Brown. The McGraw-Hill Book Company, New York, 1950. x + 498 pp. 226 figs. 16 tables. 16 × 23.5 cm. $6.50.**

THIS book provides an excellent survey of atomic physics. The author is concerned with ideas rather than with experimental or mathematical techniques. Equations are used, and the proofs, sketched, as they are needed to develop the ideas. Experiments are likewise given in outline. The book, after a brief introductory chapter, reviews subjects like Dalton's laws much as is done in a good course in general chemistry, but this doesn't prevent the author from discussing the relativity correction to electron mass as it is needed. All theories and experiments which illuminate our ideas on atoms, ions, electrons, nuclei, and protons are drawn on in this chapter. In the very readable 124 pages devoted to spectroscopy, there is much use of vector addition. The outlook here is not unlike that in Sommerfeld's classical book.

Quantum mechanics is developed briefly with considerable attention to the uncertainty principle, Fermi statistics, and the general philosophical implications. Nuclear physics gets crowded into 100 pages. The intimate connection between nuclear reactions, the heat of stars, and the birth of the universe are duly examined.

Next, we have a treatment of molecules—largely diatomic—and a final chapter on solids. These are good introductions to the fields treated.

"Atomic Physics" deserves to be read widely by those seeking an overview of this fascinating field.

HENRY EYRING

UNIVERSITY OF UTAH
SALT LAKE CITY, UTAH

● MANUAL OF STANDARDIZED PROCEDURES FOR SPECTROPHOTOMETRIC CHEMISTRY

Harold J. Fister, **Director, Viola Mae Fister Memorial Research Laboratory. Standard Scientific Supply Corporation, New York, 1950. 728 pp. Illustrated. 26.5 × 29.5 cm.**

THIS is a voluminous, practical laboratory manual containing detailed directions for carrying out some 200 clinical analytical methods, on urine, blood, feces, etc. It is intended to be a suitable set of instructions to laboratory workers with limited experience in spectrophotometry. Printed clearly, on heavy paper, and bound in a heavy loose-leaf binder, it is well designed for use on the laboratory desk.

The author lays great emphasis upon use of calibrated glassware, careful preparation and preservation of reagents, and strict adherence to the details of techniques. He gives assurance that when all this is done others will be able to duplicate his own results with these methods. He gives calibration curves for practically every method, prepared with standards in the Coleman Universal Spectrophotometer. Although it is intended that these shall be used by others only as guides, nevertheless the author is confident that if proper precautions are observed they will serve within the limits of routine clinical work.

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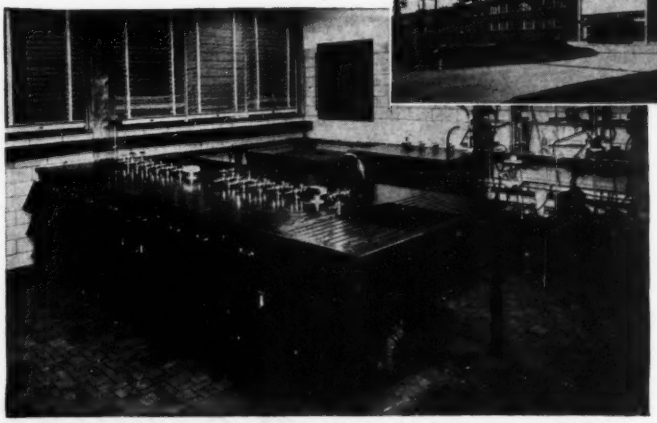

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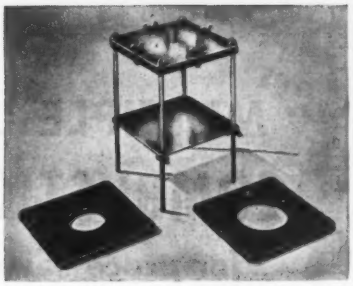

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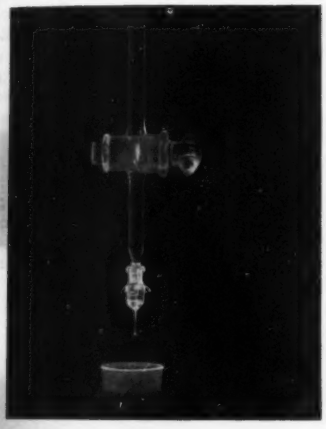

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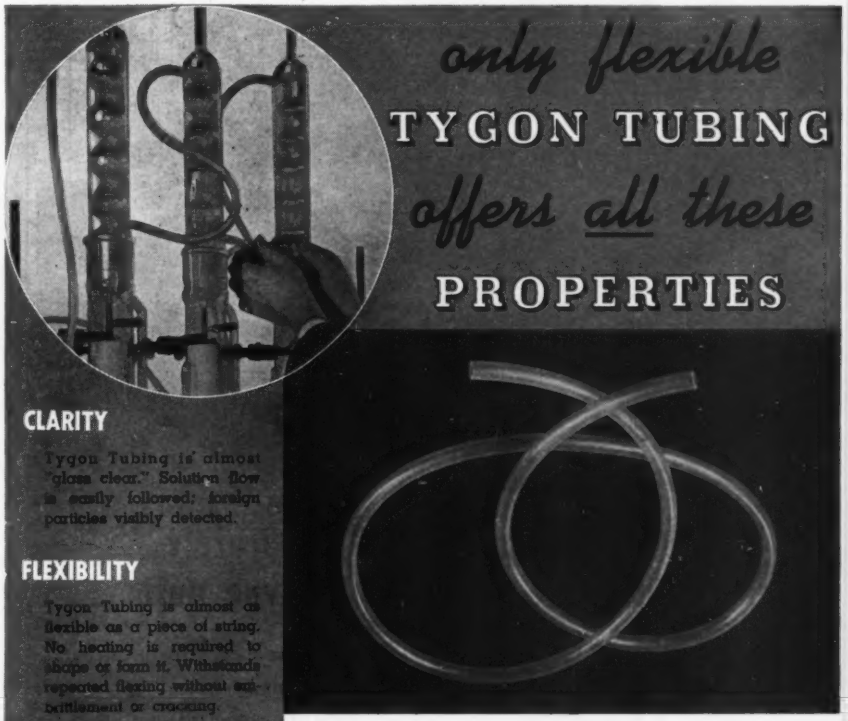
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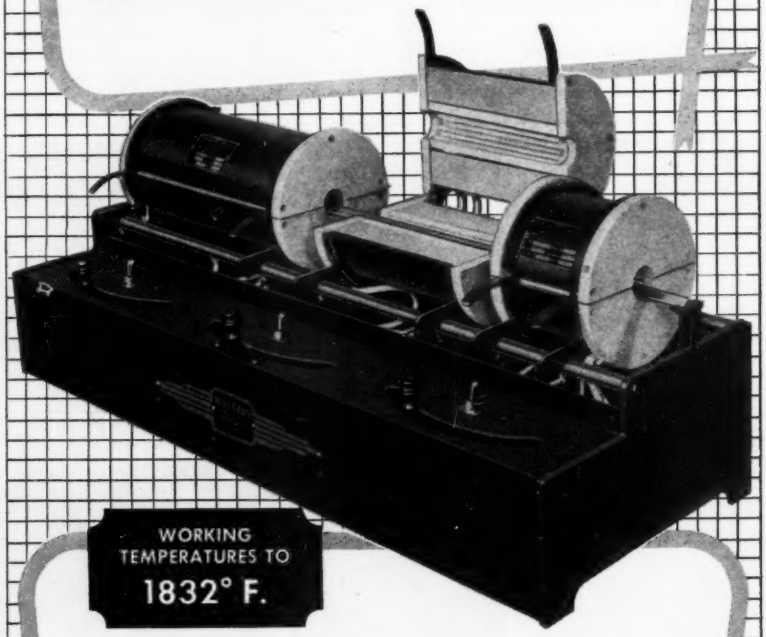

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Permanganate Cell

Mr. G. K. Venkataramaiah of Gargeswari P. O., (Via) Mysore, South India, has found that the substitution of a moderately strong solution of potassium permanganate for the common potassium dichromate solution as a deoxidizer in a zinc-carbon primary cell provides a higher voltage. The permanganate cell supplies a steady current for a long period of continuous use.

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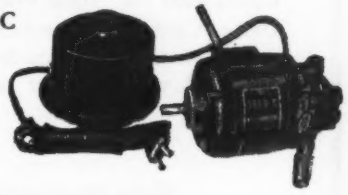

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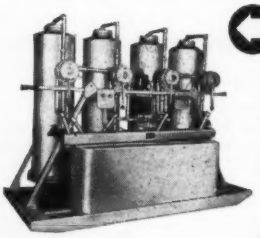

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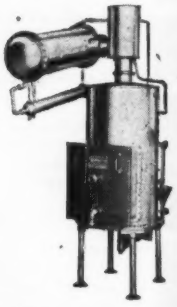

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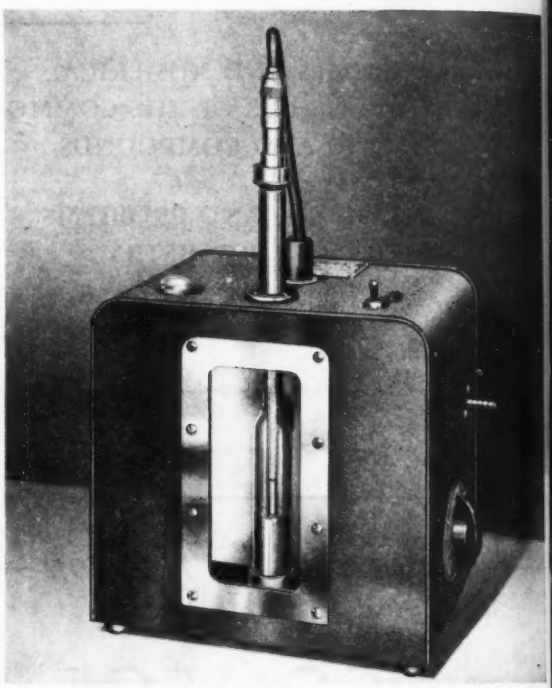

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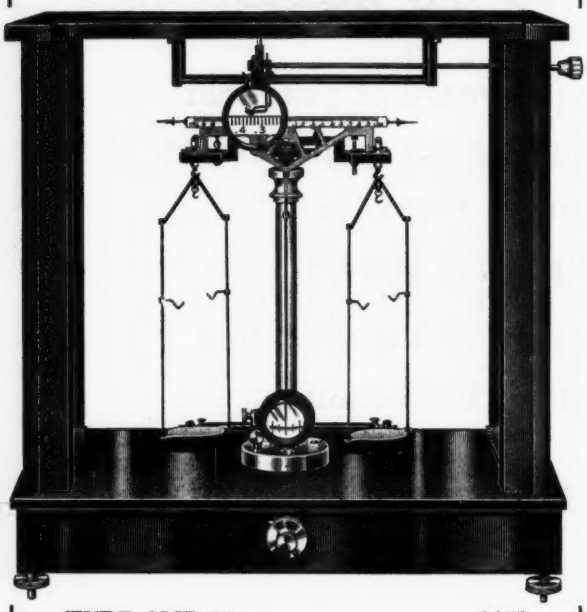

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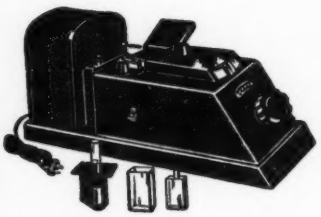

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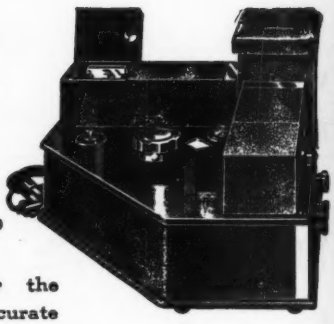

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References

(1) Ind. Eng. Chem. 25-653 (June, 1933)

(2) Ind. Eng. Chem. 25-1112 (Oct., 1933)

(3) National Bureau of Standards Journal of Research 12-241 (Feb. 1934, R.P. No. 649)

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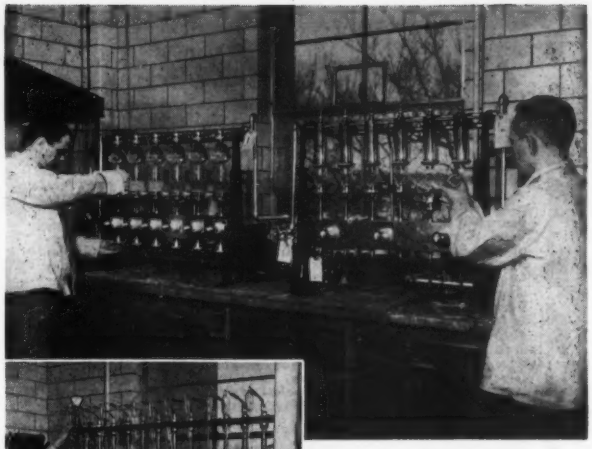

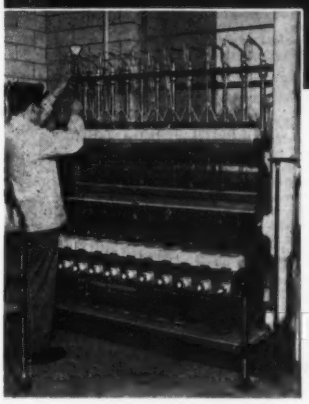

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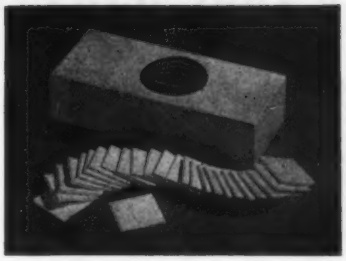

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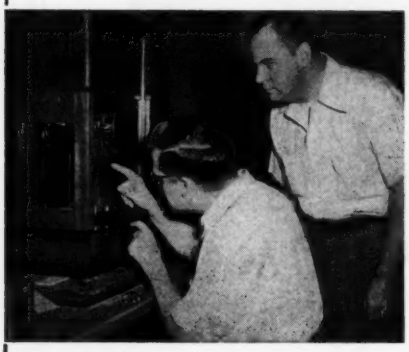

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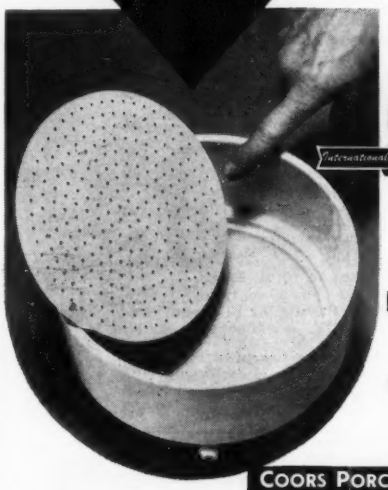

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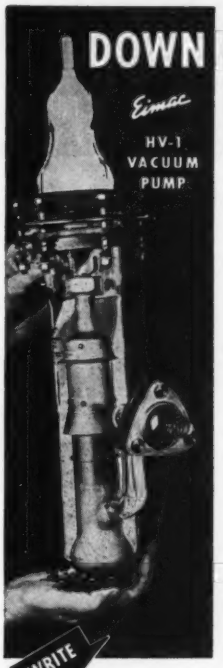

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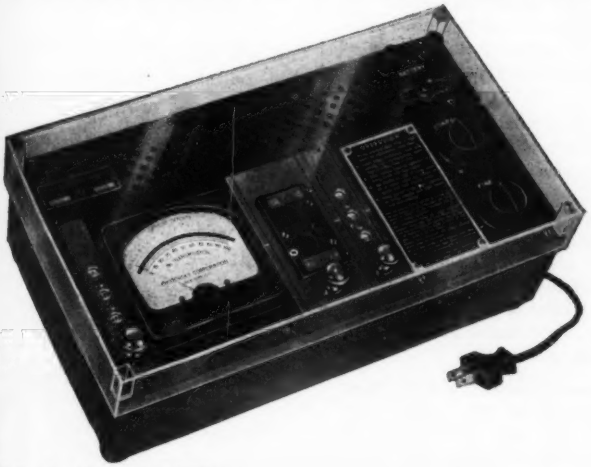

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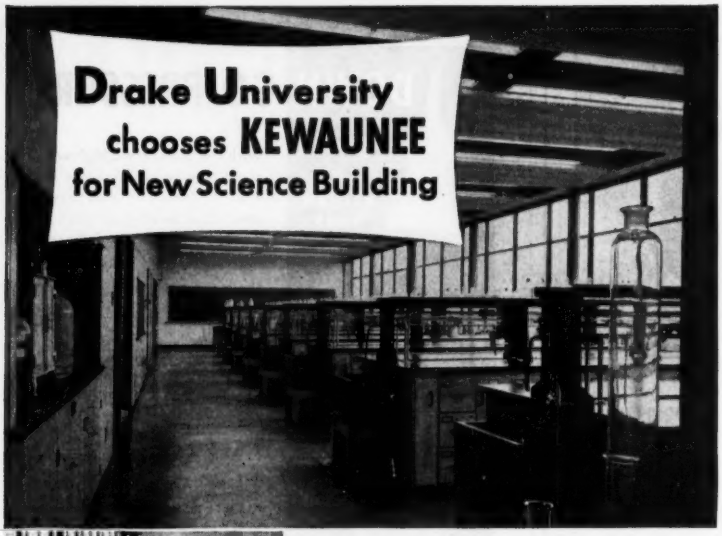

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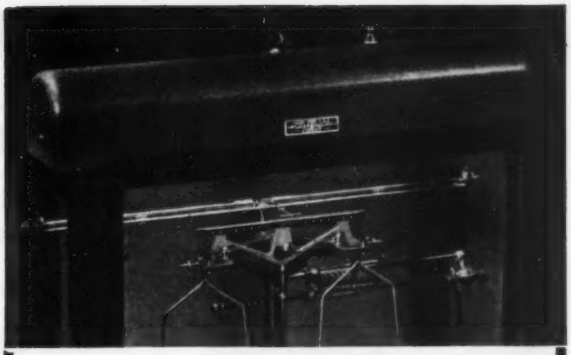

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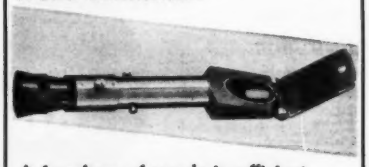

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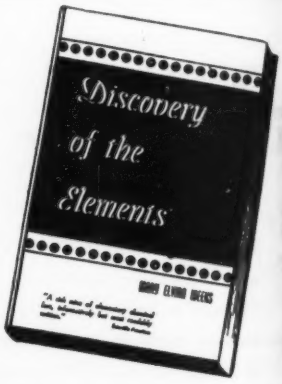

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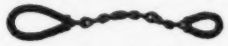

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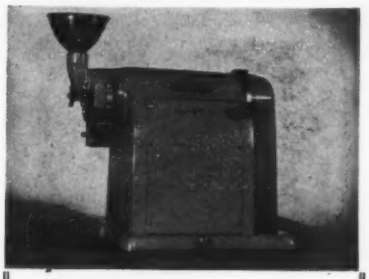

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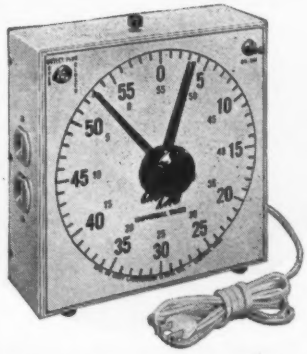

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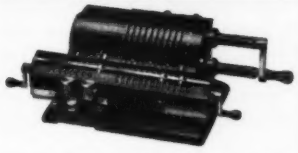

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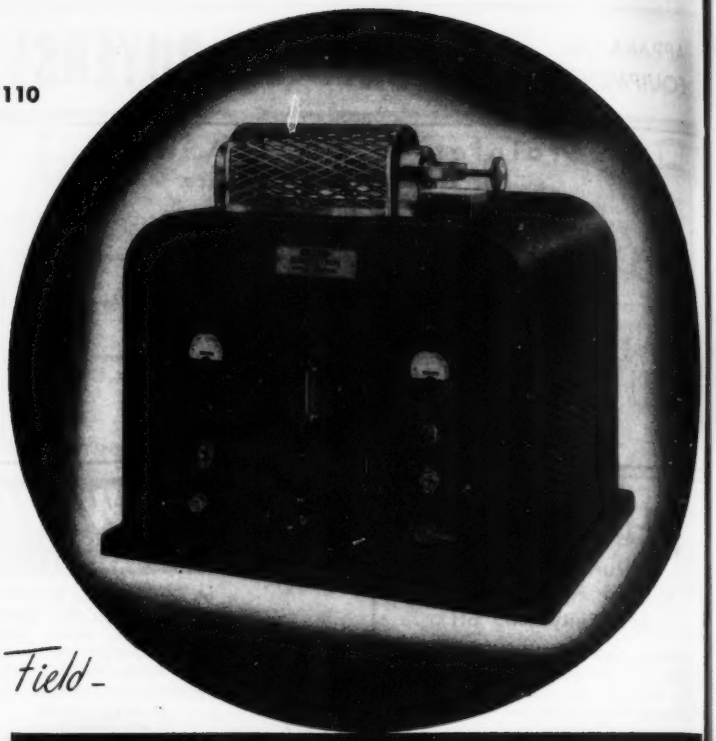

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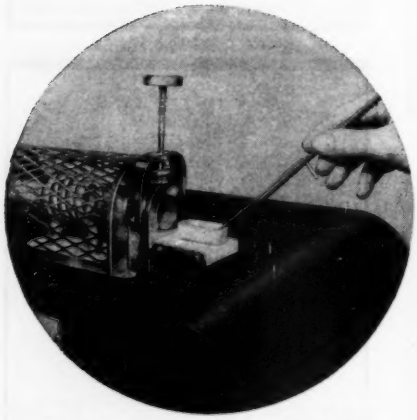

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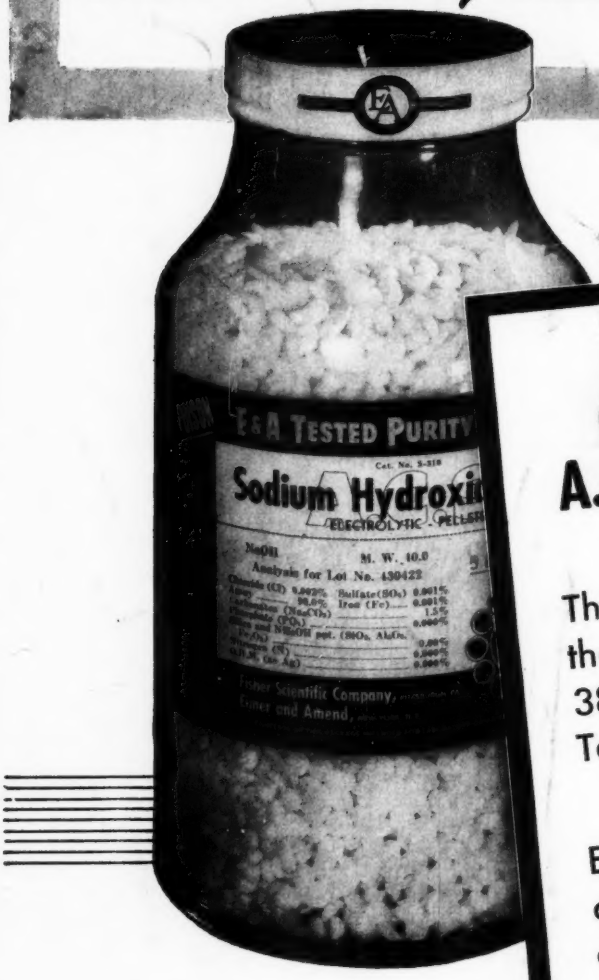

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